



**AUTHORIZATION TO DISCHARGE UNDER THE
ALASKA POLLUTANT DISCHARGE ELIMINATION SYSTEM
FOR
OIL AND GAS EXPLORATION FACILITIES IN STATE WATERS IN
COOK INLET**

GENERAL PERMIT NUMBER AKG315100

**ALASKA DEPARTMENT OF ENVIRONMENTAL CONSERVATION
Wastewater Discharge Authorization Program
555 Cordova Street
Anchorage, AK 99501**

In compliance with the provisions of the Clean Water Act (CWA), 33 U.S.C. §1251 et seq., as amended by the Water Quality Act of 1987, P.L. 100-4, this permit is issued under provisions of Alaska Statutes (AS) 46.03; the Alaska Administrative Code (AAC) as amended; and other applicable State laws and regulations.

Owners and operators of facilities engaged in oil and gas exploration, located in Cook Inlet within Offshore and Coastal Subcategories of the Oil and Gas Extraction Point Source Category (40 CFR 435, Subparts A and D) are authorized to discharge to waters of the United States, only in accordance with effluent limits, monitoring requirements, and other conditions set forth herein.

**A COPY OF THIS GENERAL PERMIT
MUST BE KEPT AT THE SITE WHERE DISCHARGES OCCUR.**

This permit is effective **Pending**.

This permit and the authorization to discharge shall expire at midnight on **[INSERT DATE]**.

The applicant shall reapply for a permit reissuance on or before **[INSERT DATE]**, 90 days before the expiration of this permit.

DRAFT

Signature

Printed Name

Date

Program Manager

Title

TABLE OF CONTENTS

| | |
|---|-----|
| SCHEDULE OF SUBMISSIONS | 4 |
| 1.0 PERMIT COVERAGE | 6 |
| 1.1 Coverage and Eligibility | 6 |
| 1.2 Notice of Intent Review and Permit Coverage Determination Process | 6 |
| 1.3 Authorized Discharges | 8 |
| 1.4 Prohibitions | 8 |
| 1.5 Requiring an Individual Permit | 9 |
| 1.6 Notification Requirements | 10 |
| 1.7 Permit Expiration | 11 |
| 2.0 LIMITS AND MONITORING REQUIREMENTS | 12 |
| 2.1 Requirements for all Discharges | 12 |
| 2.2 Requirements for Drilling Fluids and Drill Cuttings (Discharge 001) | 13 |
| 2.3 Requirements for Deck Drainage (Discharge 002) | 23 |
| 2.4 Requirements for Domestic Wastewater (Discharge 003) | 24 |
| 2.5 Requirements for Graywater (Discharge 004) | 25 |
| 2.6 Requirements for Miscellaneous Discharges (Discharges 005-013) | 26 |
| 2.7 WET Testing Requirements | 28 |
| 2.8 Reporting of Monitoring Requirements | 31 |
| 2.9 Mixing Zone and Modification of Effluent Limits | 32 |
| 2.10 Best Management Practices Plan | 33 |
| 2.11 Removed Substances | 36 |
| 3.0 SPECIAL CONDITIONS | 37 |
| 3.1 Quality Assurance Project Plan | 37 |
| 3.2 Corrective Action Plans | 37 |
| APPENDIX A STANDARD CONDITIONS | A-1 |
| APPENDIX B ACRONYMS | B-1 |
| APPENDIX C DEFINITIONS | C-1 |
| APPENDIX D COOK INLET COVERAGE AREA | D-1 |
| APPENDIX E SPP METHOD | E-1 |
| APPENDIX F PROTOCOL FOR THE DETERMINATION OF DEGRADATION OF NON-AQUEOUS BASED FLUIDS IN A MARINE CLOSED BOTTLE BIODEGRADATION TEST SYSTEM: MODIFIED ISO 11734 | F-1 |
| APPENDIX G DETERMINATION OF CRUDE OIL CONTAMINATION IN NON-AQUEOUS DRILLING FLUIDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY (GC/MS) | G-1 |

| | |
|--------------------|------------------------------|
| ATTACHMENT 1 | NOTICE OF INTENT INFORMATION |
| 1-1 | |

| | |
|--------------------|----------------------------|
| ATTACHMENT 2 | NONCOMPLIANCE NOTIFICATION |
| 2-1 | |

LIST OF TABLES

| | |
|---|------|
| Table 1: Schedule of Submissions | 4 |
| Table 2: Effluent Limitations and Monitoring Requirements for Drilling Fluids and Drill Cuttings (Discharge 001)..... | 14 |
| Table 3: Effluent Limitations and Monitoring Requirements Deck Drainage (Discharge 002)..... | 23 |
| Table 4: Effluent Limitations and Monitoring Requirements for Domestic Wastewater (Discharge 003) | 24 |
| Table 5: Effluent Limitations and Monitoring Requirements for Graywater (Discharge 004) | 25 |
| Table 6: Effluent Limitations and Monitoring Requirements for Miscellaneous Discharges 005-013 | 26 |
| Table 7: WET Trigger Levels for Surface Discharges | 27 |
| Table 8: WET Trigger Levels for Submerged Pipe Discharges | 28 |
| Table E- 1: Conditions ¹ for conducting 96 hour NAF and 10-day SBM sediment toxicity test with <i>L plumulosus</i> | E-10 |
| Table E- 2: Test acceptability requirements ¹ for 10-day NAF and 96 hour SBM test with <i>L plumulosus</i> | E-11 |
| Table E- 3: Culture conditions for <i>L plumulosus</i> | E-12 |
| Table F- 1: Test Acceptability Criteria | F-9 |
| Table G- 1: Gas Chromatograph/Mass Spectrometer (GS/MS) Operating Conditions..... | G-10 |
| Table G- 2: Approximate Retention Times for Compounds | G-10 |
| Table G- 3: Recommended Ion Mass Numbers..... | G-13 |

SCHEDULE OF SUBMISSIONS

The Schedule of Submissions summarizes some of the required submissions and activities the applicant must complete and/or submit to the Alaska Department of Environmental Conservation (DEC or the Department) during the term of this permit. All deadlines for the Schedule of Submissions are based on the postmark date of the submittal. The applicant is responsible for all submissions and activities even if they are not summarized below.

Table 1: Schedule of Submissions

| Permit Part | Submittal | Frequency | Due Date | Submit Documentation To* |
|------------------|---|----------------|--|--------------------------------------|
| 1.1.2 and 1.6 | Notice of Intent (NOI) to discharge from a new, recommencing, or expanding facility | 1/permit cycle | 45 days prior to discharge | Permitting – Appendix A, Part 1.1.1. |
| | NOI for an existing discharge authorized under the Environmental Protection Agency-issued AKG315000 | 1/permit cycle | By [To Be Determined (TBD)] | Permitting |
| 1.1.2 and 1.6.2 | Plans and Report supporting Request for Waiver from Minimum Treatment requirements, or documentation of previous plan and waiver approvals. | 1/permit cycle | With submittal of NOI (Attachment 1) | Permitting – Appendix A, Part 1.1.1. |
| 1.7 | NOI for an authorized applicant if the applicant intends to continue operations and discharges beyond the term of this permit | 1/permit cycle | 90 days before expiration of the general permit | Permitting – Appendix A, Part 1.1.1. |
| 2.10 | Best Management Practices (BMP) Plan | 1/permit cycle | BMP Plan shall be ready to implement upon submittal of an NOI, the applicant shall certify in writing that the BMP Plan has been prepared and is ready to implement prior to initial discharge | Compliance – Appendix A, Part 1.1.1. |
| 2.2.8.2 | Environmental Study Plan | 1/permit cycle | The Environmental Study Plan shall be submitted with, or prior to, a complete NOI. | Permitting |
| | Environmental Reports | 1/year/site | 180 Days following final sample collection. | Compliance |
| | End-of-Well Report | 1/Well | 90 Days following well completion | Compliance |
| Appendix A 3.4.1 | Oral notification of noncompliance | As Necessary | Within 24 hours from the time the applicant | Compliance – Appendix A, Part |

| Permit Part | Submittal | Frequency | Due Date | Submit Documentation To* |
|--|--|--------------|---|--------------------------------------|
| | | | becomes aware of the circumstances of noncompliance | 1.1.2. |
| Appendix A 3.4.1 | Written documentation of noncompliance | As Necessary | Within five days after the applicant becomes aware of the circumstances | Compliance – Appendix A, Part 1.1.2. |
| * See Appendix A Part, 1.1 for addresses | | | | |

1.0 PERMIT COVERAGE

1.1 Coverage and Eligibility

- 1.1.1 Existing facilities for oil and gas exploration in Cook Inlet that have obtained coverage under the 2007 Environmental Protection Agency (EPA) issued AKG-31-5000 General Permit (2007 Permit): Owners or operators of facilities with administratively extended coverage under the 2007 Permit will be granted coverage upon submitting an updated and complete Notice of Intent (NOI) and documentation wastewater plan and (if applicable) waiver approvals in accordance with 18 AAC 72. See Section 1.6 for notification requirements.
- 1.1.2 New facilities for oil and gas exploration with discharges within state waters in Cook Inlet that meet the criteria for coverage under this permit will be granted coverage upon submittal of a complete NOI in accordance with 18 AAC 83.210(b), treatment system plan review per 18 AAC 72.200 and 72.600, and (if applicable) a waiver request for minimum treatment requirements per 18 AAC 72.060. See Permit Part 1.6 for notification requirements.
- 1.1.3 An applicant eligible for a waiver of the secondary treatment requirements under 18 AAC 72.050(a)(4) will be authorized to discharge graywater under this permit. Discharged graywater must meet primary treatment standards (as defined under 18 AAC 72.990(50)(B)). See Permit Part 1.2 for NOI submittal requirements.
- 1.1.4 An applicant may request a mixing zone authorization from DEC by completing the mixing zone section of the NOI
- 1.1.5 Authorization to discharge requires written notification from the Department that coverage has been granted, that a mixing zone has been authorized (if applicable), and that a specific permit number has been assigned to the operation.

1.2 Notice of Intent Review and Permit Coverage Determination Process

- 1.2.1 An applicant must submit a complete NOI form (See ATTACHMENT 1) and BMP certification along with plans, reports, and (if applicable) a request to waive the minimum treatment requirements associated with 18 AAC 72.050.
- 1.2.2 An existing permittee must submit a complete NOI form (See ATTACHMENT 1) and BMP certification along with reports and documentation of approved plans and (if applicable) a waiver for minimum treatment requirements associated with 18 AAC 72.050.
- 1.2.3 The Department will review a NOI for completeness and accuracy. If an NOI is found to be incomplete, the Department will notify the applicant of the needed changes to the NOI submittal.

- 1.2.4 Prior to discharging to waters of the United States, a new applicant must submit plans for approval per 18 AAC 72.200 and/or 18 AAC 72.600. The plans shall be prepared by a licensed Alaska engineer and submitted with the NOI and be accompanied by the appropriate fee required by 18 AAC 72.955.
- 1.2.5 Under 18 AAC 72.060, an applicant may request a waiver from the requirements of 18 AAC 72.050(a)(4) by submitting a report in accordance with 18 AAC 72.050(d)(1)-(5) and 18 AAC 72.060(b). The report shall be prepared by a licensed Alaska engineer, submitted with the NOI, and be accompanied by the appropriate fee required by 18 AAC 72.955.
- 1.2.6 The Department will review the engineering plans and report to determine if the request for waiver of minimum treatment requirements of 18 AAC 72.050 should be granted and if the applicant is eligible for coverage under this permit. This determination will be made prior to authorization under this permit.
- 1.2.7 The Department will make a determination regarding the appropriateness of granting permit coverage at a proposed discharge location or area of operation.
 - 1.2.7.1 Location coordinates provided in the NOI for each proposed discharge location or area of operation will be used to determine if a discharge is prohibited by this permit or would require application for an Alaska Pollutant Discharge Elimination System (APDES) individual permit.
- 1.2.8 The Department will review the applicant submittal to authorize a standard sized cylindrically shaped 100 meter mixing zone for specific discharges and parameters per Section 2.9.
 - 1.2.8.1 When authorizing a standard size cylindrically shaped 100 meter mixing zone, the Department will consider whether the discharges requested are consistent with permit conditions.
- 1.2.9 Upon completion of the NOI review, the Department will either:
 - 1.2.9.1 Prepare and transmit a written coverage determination specifying whether (1) The information required by 18 AAC 72.050(d)(1) – (5) is sufficient to waive minimum treatment requirements required by 18 AAC 72.050, and (2) a standard size cylindrically shaped mixing zone is authorized per Section 2.9; or
 - 1.2.9.2 Notify the applicant of needed changes to the NOI submittal; or
 - 1.2.9.3 Deny coverage under the permit and require an applicant to submit an individual permit application.

1.3 Authorized Discharges

- 1.3.1 This permit authorizes and places conditions on discharges from oil and gas exploration facilities that are located within Cook Inlet (see Figure 1) under the Offshore and Coastal Subcategories of the Oil and Gas Extraction Point Source Category (40 CFR Part 435, Subparts A and D) as adopted by reference at 18 AAC 83.010(g)(3).
- 1.3.2 This permit authorizes the following discharges from facilities related to oil and gas exploration:

| <u>Discharge Number</u> | <u>Discharge Description</u> |
|-------------------------|---|
| 001 | Drilling Fluids and Drill Cuttings |
| 002 | Deck Drainage |
| 003 | Domestic Wastewater |
| 004 | Graywater |
| 005 | Desalination Unit Wastes |
| 006 | Blowout Preventer Fluid |
| 007 | Boiler Blowdown |
| 008 | Fire Control System Test Water |
| 009 | Non-Contact Cooling Water |
| 010 | Uncontaminated Ballast Water |
| 011 | Bilge Water |
| 012 | Excess Cement Slurry |
| 013 | Mud, Cuttings, and Cement at the Seafloor |

1.4 Prohibitions

- 1.4.1 This permit prohibits the discharge of any waste streams, including spills and other unintentional or non-routine discharges of pollutants that are not part of the normal operation of the facility.
- 1.4.2 This permit prohibits the discharge to any receiving water that is listed on the Clean Water Act (CWA) Section 303(d) list as impaired for failure to meet a water quality standard (WQS) and the facility discharges a pollutant that causes or contributes to the impairment.
- 1.4.3 This permit prohibits the discharge of any pollutant that is not expressly authorized in the permit.
- 1.4.4 This permit prohibits discharges shoreward of the 10 meter mean lower low water (MLLW) isobaths.
- 1.4.5 An applicant should contact DEC if there is uncertainty whether discharges will be located in a prohibited area. This permit prohibits discharges to the following areas:

1.4.5.1 Within the boundaries or within 4,000 meters of a coastal marsh, river delta, river mouth, designated State Game Refuge, State Game Sanctuary, Critical Habitat Area, National Park, or Area Meriting Special Attention (ASMA). The seaward edge of a coastal marsh is defined as the seaward edge of emergent wetland vegetation.

1.4.5.1.1 The following are located in the vicinity of the permit coverage area:

| | |
|------------------------|--|
| State Game Refuge: | Susitna Flats Trading Bay |
| Critical Habitat Area: | Kalgin Island Redoubt Bay Clam Gulch Kachemak Bay |
| National Park: | Lake Clark |
| ASMA: | Port Graham/Nanwalek |

1.4.5.1.2 The legal descriptions of state specialty areas are found in AS 16.20 Conservation and Protection of Alaska Fish and Game. Further Information can be obtained from the Alaska Department of Fish and Game, 333 Raspberry Road, Anchorage, AK 99501; phone (907) 237-2342.

1.4.5.2 In Kamishak Bay, west of the line from Cape Douglas to Chinitna Point.

1.4.5.3 In Chinitna Bay, inside of the line between the points of the shoreline at latitude 59°52'45" N, longitude 152°48'18" W on the north and latitude 59°46'12" N, longitude 153°00'24" W on the south (Figure 1).

1.4.5.4 In Tuxedni Bay, inside of the lines on either side of Chisik Island (Figure 1).

1.4.5.4.1 From latitude 60°04'06" N, longitude 152°34'12" W on the mainland to the southern tip of Chisik Island (latitude 60°05'45" N, longitude 152°33'30" W).

1.4.5.4.2 From the point on the mainland at latitude 60°13'45" N, longitude 152°32'42" W to the point on the north side of Snug Harbor on Chisik Island (latitude 60°06'36" N, longitude 152°32'54" W).

1.4.5.5 Within tracts identified in the Alaska Department of Natural Resources, Oil and Gas Division's Mitigation Measure Analysis: Cook Inlet, revised March 2012.

1.5 Requiring an Individual Permit

1.5.1 The Department may require an applicant authorized to discharge under a general permit to apply for and obtain coverage under an individual permit, or any interested person may petition the Department to take this action.

- 1.5.2 The Department will notify the applicant in writing by certified mail that an individual permit application is required. If an applicant fails to submit an individual permit application by the date required in the notification, coverage under this permit is automatically terminated at the end of the day specified for application submittal.
- 1.5.3 An applicant authorized by this permit may request to be excluded from the coverage of this permit by applying for an individual permit. The applicant shall submit an individual permit application (APDES permit application Form 1 and Form 2C and Form 2M if applicable) with reasons supporting the request to the Department at the address in Appendix A, Part 1.1.1.
- 1.5.4 When an individual permit is issued to an applicant otherwise covered by this permit, the applicability of this permit to the applicant is automatically terminated on the effective date of the individual permit.
- 1.5.5 When an individual permit is denied to an applicant otherwise covered by this permit, the applicant is automatically reinstated under this permit on the date of such denial, unless the applicant cannot meet the conditions of the permit or otherwise specified by the Department.
- 1.5.6 An applicant excluded from this permit solely because the applicant already has an individual permit may request that the individual permit be revoked and that the applicant be covered by this permit. Upon revocation of the individual permit, and if the applicant can comply with the terms of this permit, this permit shall apply to the applicant.

1.6 Notification Requirements

- 1.6.1 To obtain permit coverage as a first time permittee of a facility eligible for this permit, the applicant shall submit plans and reports necessary to obtain approval to discharge and receive approval of a waiver from minimum treatment requirements if applicable. The information shall be submitted with a complete NOI and report supporting the request for a waiver from minimum treatment requirements as described in ATTACHMENT 1 of this permit.
 - 1.6.1.1 Notification must be made 45 days prior to discharge from a new, recommencing, or expanded facility.
- 1.6.2 An existing permittee who submitted an NOI prior to the expiration date of the EPA-issued 2007 permit AKG-31-5000 is required to submit a new NOI along with a documentation demonstrating previous approval of plan review and waiver from minimum treatment requirements under 18 AAC 72.060, if applicable.
 - 1.6.2.1 Notification must be made prior to **[Date TBD]**..

- 1.6.3 The NOI shall be signed by the owner, or other signatory authority, in accordance with Appendix A, Part 1.12 (Signature Requirements), and a copy must be retained on site in accordance with Appendix A, Part 1.11 (Monitoring and Records).
- 1.6.4 A mixing zone request may be included with the NOI.
- 1.6.5 The Department will authorize a standard size cylindrically shaped 100 meter radius mixing zone centered over the outfall pipe or discharge pipe terminus and extending from the sea floor to the sea surface.
- 1.6.6 The applicant must submit a NOI to DEC at the address in Appendix A, Part 1.1.1.

1.7 Permit Expiration

This permit will expire at midnight on **[Date TBD]**. A permittee wishing to continue coverage under a reissued permit must submit a new NOI at least 90 days prior to the expiration of this permit, as described in Standard Conditions, Appendix A, Part 1.3.

2.0 LIMITS AND MONITORING REQUIREMENTS

2.1 Requirements for all Discharges

- 2.1.1 During the effective period of this permit, the permittee is authorized to discharge pollutants within the area of coverage set forth in Sections 1.4 and Appendix D, in accordance with the limits and conditions set forth herein.
- 2.1.2 This permit authorizes the discharge of only those pollutants resulting from facility processes, waste streams, and operations that have been clearly identified in the NOI.
- 2.1.3 The permittee must collect all effluent samples from the effluent stream of each discharge after the last treatment unit prior to discharge into the receiving waters, except as otherwise required by discharge-specific sections of this permit.
- 2.1.4 The permittee must comply with the effluent limits in this permit at all times unless otherwise indicated, regardless of the frequency of monitoring or reporting required by other provisions of this permit.
- 2.1.5 Unless specifically addressed in this permit, the permittee shall not discharge free oil, floating solids, debris, sludge, deposits, foam, scum, or other residues of any kind.
- 2.1.6 The permittee must minimize the discharge of surfactants, dispersants, and detergents except as necessary to comply with the safety requirements of the Occupational Health and Safety Administration. This restriction applies to tank cleaning and other operations that do not directly involve the safety of workers. The permittee must report all discharge of surfactants, dispersants, and detergents with the monthly discharge monitoring report (DMR). The discharge of dispersants to marine waters in response to oil or other hazardous waste spills is not authorized.
- 2.1.7 The permittee must separate area drains for washdown and rainfall that may be contaminated with oil and grease from those area drains that would not be contaminated so that the waste streams are not comingled. Deck drainage that is contaminated with oil and grease must be processed through an oil-water separator prior to discharge.
- 2.1.8 The permittee is not required to conduct monitoring for the facility when it is not staffed and no discharges are commencing. The permittee must provide DEC written notification that the facility is no longer staffed at least 30 days prior to terminating monitoring requirements.
- 2.1.9 The permittee shall not discharge diesel oil, halogenated phenol compounds, trisodium nitrilotriacetic acid, sodium chromate, or sodium dichromate.

- 2.1.10 If any discharges are comingled, the most stringent effluent limit for each individual discharge shall be applied to the resulting discharge. If the individual discharge is not authorized, the comingled discharge is not authorized. Monitoring for compliance with technology based effluent limits must be accomplished prior to commingling.
- 2.1.11 If requested, the permittee must provide DEC with a sample of any waste stream in the manner specified by DEC as soon as practicable after the request.
- 2.1.12 The discharge of maintenance waste such as removed paint and materials associated with surface preparation and coating application is prohibited. Such materials must be contained to the maximum extent practicable using vacuum abrasive blasting, covering grated areas with plywood, surrounding the area with canvas tarps, and similar measures to capture as much material as practicable. All collected material must be disposed of at an appropriate shore based facility. Prior to conducting sandblasting or similar maintenance activities, the permittee must develop and implement a BMP Plan for the containment of waste material.

2.2 Requirements for Drilling Fluids and Drill Cuttings (Discharge 001)

- 2.2.1 The discharge of drilling fluids and drill cuttings is only authorized at exploratory facilities.
- 2.2.2 The discharge of non-aqueous based drilling fluids is prohibited except for situations where such fluids adhere to drill cuttings at facilities within the Territorial Seas, as defined 40 CFR Part 435.
- 2.2.3 In addition to requirements in Section 2.1, the permittee must comply with the following effluent limitations and monitoring requirements:

Table 2: Effluent Limitations and Monitoring Requirements for Drilling Fluids and Drill Cuttings (Discharge 001)

| Discharge | Pollutant Parameter | Effluent Limitations | Monitoring Requirements | |
|---|---|--|--------------------------------------|-------------|
| | | Average Monthly and Maximum Daily Limits | Measurement Frequency | Sample Type |
| Water-based fluids and cuttings | Suspended Particulate Phase toxicity ¹ | Minimum 96-hour LC ₅₀ of 30,000 ppm | Monthly and End-of-Well ² | Grab |
| | Free oil | No discharge ³ | Daily | Grab |
| | Diesel oil ⁴ | No discharge | Daily | Grab |
| | Mercury | 1 mg/kg ⁵ | Once per well | Grab |
| | Cadmium | 3 mg/kg ⁵ | Once per well | Grab |
| | Volume (MG) | Report average and maximum daily and monthly total | Monthly | Estimate |
| | Depth Dependent Discharge Rate 0 to 5 meters >5 to 20 meters >20 to 40 meters >40 meters | No discharge 500 barrels (bbl)/hr 750 bbl/hr 1,000 bbl/hr | Continuous during discharge | Estimate |
| Non-aqueous fluids | Drilling fluids | No discharge | Daily | Observation |
| Non-aqueous stock base fluid (C ₁₆ -C ₁₈ internal olefin, C ₁₂ -C ₁₄ ester or C ₈ ester) | Mercury | 1 mg/kg ⁵ | Annual | Grab |
| | Cadmium | 3 mg/kg ⁵ | Annual | Grab |
| | Polynuclear Aromatic Hydrocarbons (PAH) | mass ratio ⁶ < 1x10 ⁻⁵ | Annual | Grab |
| | Sediment toxicity | ratio ⁷ < 1.0 | Annual | Grab |
| | Biodegradation rate | ratio ⁸ < 1.0 | Annual | Grab |
| | Volume (MG) | Report average and maximum daily and monthly total | Monthly | Estimate |
| Non-Aqueous Drilling Fluids which adhere to drill cuttings (Territorial Seas Only per 40 CFR Part 435) ¹⁵ | Free Oil | No discharge ³ | Daily | Grab |
| | Diesel oil ² | No discharge | Daily | Grab |
| | SPP toxicity ¹ | Minimum 96-hour LC ₅₀ of 30,000 ppm | Monthly and End-of-Well ² | Grab |
| | Sediment toxicity | Drilling fluid sediment toxicity ratio ⁹ < 1.0 | Annual | Grab |
| | Formation oil | No discharge ¹⁰ | Daily | Grab |
| | Base fluid retained on drill cuttings (C ₁₆ -C ₁₈ internal olefin stock ¹¹) | 6.9 g NAF base fluid/100 g wet drill cuttings ¹² | Daily ¹³ | Grab |
| | Base fluid retained on drill cuttings ¹⁴ | 9.4 g NAF base Fluid/100 g wet drill cuttings ¹² | Daily ¹³ | Grab |

| Discharge | Pollutant Parameter | Effluent Limitations | Monitoring Requirements | |
|-----------|--|--|-------------------------|-------------|
| | | Average Monthly and Maximum Daily Limits | Measurement Frequency | Sample Type |
| | (C ₁₂ -C ₁₄ ester or C ₈ ester stock) | | | |
| | Volume (MG) | Report average and maximum daily and monthly total | Monthly | Estimate |

Footnotes:

1. As determined by the 96-hour suspended particulate phase (SPP) toxicity test. See 40 CFR 435, Subpart A, Appendix 2.
2. At the end-of-well, a sample must be collected for toxicity testing where no mineral oil is used. This sample can also serve as the monthly monitoring sample
3. As determined by the Static Sheen Test. See 40 CFR Part 435, Subpart A, Appendix 1.
4. See Section 2.2.7.3.
5. Dry weight in the stock barite. Analysis shall be conducted using EPA Methods 245.5 or 7471b for mercury and 200.7 for cadmium. The permittee shall analyze a representative sample of stock barite once prior to drilling each well and submit the results with the DMR for the month in which drilling operations commence for the respective well. If the permittee uses the same supply of stock barite to drill subsequent wells, the permittee may submit the same analysis for those subsequent wells. (See Section 2.2.7.6)
6. PAH mass ratio = [mass (g) of PAH (as phenanthrene)] ÷ [mass (g) of stock base fluid] as determined by EPA method 1654, Revision A, entitled "PAH Content of Oil by HPLC/UV," December 1992 (See Section 2.2.4.2).
7. Base fluid sediment toxicity ratio = [10-day LC₅₀ of C₁₆-C₁₈ internal olefin, C₁₂-C₁₄ ester or C₈ ester] ÷ [10-day LC₅₀ of stock base fluid] as determined by ASTM E 1367-92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, after preparing the sediment according to the method specified at 40 CFR 435, Subpart A, Appendix 3 (See Section 2.2.4.6). Results of up to 3 tests may be averaged to determine compliance, using 2 samples from the same lot of stock fluids. Equivalent aliquots of one homogenized sample must be split by laboratory (parts 1A and 1B) and tested separately if averaging is used. Permittees may show compliance based on test results from part 1A or from the rounded arithmetic average of the test results from part 1A and 1B. Permittees may also test the second sample for compliance. Where the second sample is analyzed, operators will determine compliance using the arithmetic average of the results from all 3 tests. Permittees shall report the appropriate number on the DMR. With the DMR, the permittee must submit documentation showing how the number was calculated and all applicable test reports (See Section 2.2.4.6).
8. Biodegradation rate ratio = [cumulative gas production (ml) of C₁₆-C₁₈ internal olefin, C₁₂-C₁₄ ester or C₈ ester] ÷ [cumulative gas production (ml) of stock base fluid], both at 275 days as determined by ISO 11734:1995 method: "Water quality - Evaluation of the 'ultimate' anaerobic biodegradability of organic compounds in digested sludge--Method by measurement of the biogas production (1995 edition)" as modified for the marine environment (See Section 2.2.4.1). Results of up to 3 tests may be averaged to determine compliance, using 2 samples from the same lot of stock fluids. Equivalent aliquots of one homogenized sample must be split by laboratory (parts 1A and 1B) and tested separately if averaging is used. Permittees may show compliance based on test results from part 1A or from the rounded arithmetic average of the test results from part

| Discharge | Pollutant Parameter | Effluent Limitations | Monitoring Requirements | |
|-----------|---------------------|--|-------------------------|-------------|
| | | Average Monthly and Maximum Daily Limits | Measurement Frequency | Sample Type |
| | | <p>1A and 1B. Permittees may also test the second sample for compliance. Where the second sample is analyzed, operators will determine compliance using the arithmetic average of the results from all 3 tests. Permittees shall report the appropriate number on the DMR. With the DMR, the permittee must submit documentation showing how the number was calculated and all applicable test reports</p> <p>9. Drilling fluid sediment toxicity ratio = [4-day LC_{50} of C_{16}-C_{18} internal olefin] ÷ [4-day LC_{50} of drilling fluid removed from drill cuttings at the solids control equipment] as determined by ASTM E 1367-92 method: "Standard Guide for Conducting 10-day Static Sediment Toxicity Tests with Marine and Estuarine Amphipods," 1992, after preparing the sediment according to the method specified in Appendix B of this permit. Results of up to 3 tests may be averaged to determine compliance, using 2 grab samples collected no more than 15 minutes apart. Equivalent aliquots of the first, homogenized sample must be split by the laboratory (parts 1A and 1B) and tested separately if averaging is used. Permittees may show compliance based on test results from part 1A or from the rounded arithmetic average of the test results from parts 1A and 1B. Permittees may also test the second sample for compliance with this limit. Where the second sample is analyzed, operators will determine compliance using the arithmetic average of the results from all 3 tests. Permittees shall report the appropriate number on the DMR. With the DMR, the permittee must submit documentation showing how the number was calculated and all applicable test reports.</p> <p>10. As determined before drilling fluids are shipped offshore by the GC/MS compliance assurance method (see Section 2.2.4.3), and as determined prior to discharge by the Reverse Phase Extraction (RPE) method (See Section 2.2.4.3) applied to drilling fluid removed from drill cuttings. If the operator wishes to confirm the results of the RPE method, the operator may use the GC/MS compliance assurance method (See Section 2.2.4.4). Results from the GC/MS compliance assurance method shall supersede the results of the RPE method.</p> <p>11. This limitation is applicable only when the NAF base fluid meets the stock limitations defined in this table.</p> <p>12. As determined by the American Petroleum Institute (API) retort method (See Section 2.2.4.5).</p> <p>13. Monitoring shall be performed at least once per day when generating new cuttings. Operators conducting fast drilling (<i>i.e.</i>, greater than 500 linear feet advancement of the drill bit per day using non-aqueous fluids) shall collect and analyze one set of drill cuttings samples per 500 linear feet drilled, with a maximum of three sets per day. Operators shall collect a single discrete drill cuttings sample for each point of discharge to the ocean. The weighted average of the results of all discharge points for each sampling interval will be used to determine compliance.</p> <p>14. Averaged over all well sections.</p> <p>15. Exemptions to the zero discharge of non-aqueous drilling fluids which adhere to drill cuttings based on technical limitations may be granted per 40 CFR Part 435, Appendix A of Subpart D Coastal Subcategory.</p> | | |

2.2.4 Specific Test Requirements

2.2.4.1 Biodegradation Rate.

The approved test method for permit compliance is identified as: modified ISO 11734:1995 method: "Water quality - Evaluation of the 'ultimate' anaerobic biodegradability of organic compounds in digested sludge - Method by measurement of the biogas production (1995 edition)" (Available from the American National Standards Institute, 11 West 42nd Street, 13th Floor, New York, NY 10036) supplemented with modifications in Appendix 4 of 40 CFR 435, Subpart A and detailed in Appendix E. Compliance with the biodegradation limit will be determined using the following ratio:

$$\frac{\% \text{ Theoretical gas production of reference fluid}}{\% \text{ Theoretical gas production of NAF}} \leq 1.0$$

Where: NAF = stock base fluid being tested for compliance

Reference Fluid = C16-C18 internal olefin or C12-C14 or C8 ester reference fluid

2.2.4.2 Polynuclear Aromatic Hydrocarbons

For analysis of TAH and TAqH all analytical requirements cited in the Alaska Standards, 18 ACC 70.020(b) are applicable.

2.2.4.3 Formation Oil Contaminated Drilling Fluids

The approved test method for permit compliance is identified as GC/MS. The GC/MS method reports results for the GC/MS test as percent crude contamination when calibrated for a specific crude oil. In order to define an applicable pass/fail limit to cover a variety of crude oils, the same crude oil used in calibration of the RPE test shall be used to calibrate the GC/MS test results to a standardized ratio of the target aromatic ION Scan 105. Based on the performance of a range of crude oils against standardized ratio, a value will be selected as a pass/fail standard which will represent detection of crude oil.

2.2.4.4 Formation Oil Contamination of Discharged Drilling Fluids Retained on Cuttings

The approved test method for permit compliance is identified as: Reverse Phase Extraction (RPE) as described in Appendix 6 of 40 CFR Part 435, Subpart A. If the operator wishes to confirm the results of the RPE method (Appendix 6 of 40 CFR Part 435, Subpart A), the operator may use the GC/MS compliance assurance method (Appendix 5 of 40 CFR Part 435, Subpart A). Results from the GC/MS compliance assurance method shall supercede the results of the RPE method (Appendix 6 of 40 CFR Part 435, Subpart A).

2.2.4.5 Retention of Non-aqueous Based Drilling Fluids on Cuttings

The approved test method for permit compliance is identified as: the Retort Test Method described in Appendix 7 of 40 CFR Part 435, Subpart A. The required sampling, handling, and documentation procedures are listed in Addendum A of 40 CFR Part 435, Subpart A, Appendix 7.

2.2.4.6 Stock Base Fluid Sediment Toxicity.

The approved test method for permit compliance is identified as: ASTM E1367–99 method Standard Guide for Conducting Static Sediment Toxicity Tests with Marine and Estuarine Amphipods (Available from the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA, 19428) with *Leptocheirus plumulosus* as the test organism and sediment preparation procedures specified in Appendix 3 of 40 CFR Part 435, Subpart A and the method found in Appendix B of this permit.

2.2.5 Number of Wells

The permittee is limited to drilling discharges from no more than five wells at a single site. If a step-out or sidetracked well is drilled from a previously drilled well hole, the step-out well is considered a new well. Requests to discharge from more than five wells per site will be considered by DEC on a case-by-case basis. The permittee may only discharge from more than five wells upon approval by DEC. The permittee must submit the following information to DEC for consideration for approval of the discharge from additional wells:

- 2.2.5.1.1 Number of additional wells;
- 2.2.5.1.2 Technical analysis of additional impacts to the receiving waters;
- 2.2.5.1.3 Drilling fluid category and group for each well; and
- 2.2.5.1.4 Well information for each additional well, including well name, number latitude, longitude, beginning drill date, and hole diameter.

2.2.6 Mineral Oil Pills

- 2.2.6.1 The discharge of residual amounts of mineral oil pills (mineral oil plus additives) is authorized by this general permit provided that the mineral oil pill and at least a 50 barrels (bbl) buffer of drilling fluid on either side of the pill are removed from the circulating drilling fluid system and not discharged to waters of the United States. If more than one pill is applied to a single well, the previous pill and buffer must be removed prior to application of a subsequent pill.
- 2.2.6.2 Residual mineral oil concentration in the discharged mud must not exceed 2 percent volume/volume (API Recommended Practice 13-1, 1990). The permittee must report the following information within 60 days of the discharge if drilling mud containing residual mineral oil pill (after pill and buffer removal) is discharged:

- 2.2.6.2.1 Dates of pill application, recovery, and discharge;
- 2.2.6.2.2 Results of the Drilling Fluids Toxicity Test on samples of the mud before each pill is added and after removal of each pill and buffer (taken when residual mineral oil pill concentration is expected to be greatest);
- 2.2.6.2.3 Name of spotting compound and mineral oil product used;
- 2.2.6.2.4 Volumes of spotting compound, mineral oil, water, and barite in the pill;
- 2.2.6.2.5 Total volume of mud circulating prior to pill application, volume of pill formulated, and volume of pill circulated;
- 2.2.6.2.6 Volume of pill recovered, volume of mud buffer recovered, and volume of mud circulating after pill and buffer recovery;
- 2.2.6.2.7 Percent recovery of the pill (include calculations);
- 2.2.6.2.8 Estimated concentrations of residual spotting compound and mineral oil in the sample of mud discharged, as determined from amounts added and total mud volume circulating prior to pill application;
- 2.2.6.2.9 Measured oil content of the mud samples, as determined by the API retort method; and
- 2.2.6.2.10 An itemization of other drilling fluid components and specialty additives contained in the discharged mud concentrations reported in gal/bbl or lbs/bbl.

2.2.7 Monitoring Requirements

- 2.2.7.1 Chemical Inventory. For each mud system discharged, the permittee must maintain material safety data sheets (MSDS) and a precise chemical inventory of all constituents added downhole, including all drilling mud additives used to meet specific drilling requirements. The permittee must maintain these records for each mud system for a period of five years, and must make these records available to DEC upon request. This information is reported as part of the end of well report described in Section 2.2.6.2.
- 2.2.7.2 End of Well Reports. The permittee is required to submit an end-of-well report within 90 days of well completion. The permittee shall report the following for each drilling fluid system in the end-of-well report:
 - 2.2.7.2.1 Well name, number, latitude, longitude, beginning drill date, and hole diameter, and well completion date;
 - 2.2.7.2.2 A precise chemical inventory of all constituents added downhole, including all drilling fluid additives used to meet specific drilling requirements;
 - 2.2.7.2.3 The base drilling fluid type;

- 2.2.7.2.4 The name and total amount of each constituent in the discharged drilling fluid;
- 2.2.7.2.5 The total volumes of drilling fluid create and added downhole;
- 2.2.7.2.6 The maximum concentration of each constituent in the drilling fluid;
- 2.2.7.2.7 The total volumes of drilling fluid discharged to surface waters; and
- 2.2.7.2.8 The estimated amount of each constituent in the drilling fluid discharged to surface waters.

2.2.7.3 Diesel Oil

- 2.2.7.3.1 Compliance with the limitation on diesel oil must be demonstrated by GC analysis of drilling muds collected from the mud used at the greatest well depth (“end-of-well” sample) and of any muds or cuttings which fail the daily Static Sheen Test. In all cases, the determination of the presence or absence of diesel oil must be based on a comparison of the fingerprint of the sample and of the diesel oil in storage at the facility. The method for analysis must be EPA SW846 Method 8015C (2007). Gas chromatography/mass spectrometry (GC/MS) may be used if an instance should arise where the permittee and DEC determine that greater resolution of the drilling mud “fingerprint” is needed for a particular drilling mud sample.
- 2.2.7.3.2 The results and raw data, including the spectra, from the GC analysis must be provided to DEC by written report (1) within 30 days of a positive result with the Static Sheen Test when a discharge has occurred, or (2) for the end-of-well analysis, within 90 days of well completion.

2.2.7.4 Static Sheen Test

- 2.2.7.4.1 The permittee must perform the Static Sheen Test on separate samples of drilling muds and cuttings, as required in 40 CFR Part 435, Subpart A, Appendix 1. Samples must be collected on each day of discharge and prior to bulk discharges.
- 2.2.7.4.2 The test must be conducted in accordance with “Approved Methodology: Laboratory Sheen Tests for the Offshore Subcategory, Oil and Gas Extraction Industry,” 40 CFR Part 435, Subpart A, Appendix 1. For discharge below ice or during periods of unstable or broken ice, water temperature for the Static Sheen Test must approximate surface water temperatures at ice breakup.
- 2.2.7.4.3 Whenever muds or cuttings fail the Static Sheen Test, and a discharge has occurred in the past 24 hours, the permittee is required to analyze an undiluted sample of the material which failed the test to determine the presence or absence of diesel oil. The determination and reporting results must be performed according to Section 2.2.3.3.

2.2.7.5 Metals Analysis

2.2.7.5.1 The permittee must analyze each discharged mud system for the following metals: barium, cadmium, chromium, copper, mercury, zinc, and lead. Analyses for total recoverable concentrations must be conducted and reported for each metal utilizing the methods specified in 40 CFR Part 136. The results must be reported in “mg/kg of whole mud (dry weight)” and the moisture content (percent by weight) of the original drilling mud sample must be included in the end-of-well report.

2.2.7.5.2 Samples must be collected when the residual mineral concentration is at its maximum value. If no mineral oil is used, the analysis must be done on a drilling mud sample from the mud system used at the greatest well depth. All samples must be collected prior to any pre-dilution.

2.2.7.6 Mercury and Cadmium Content in Barite

2.2.7.6.1 The permittee must analyze a representative sample of stock barite once prior to drilling each well and submit the results for total mercury and total cadmium in the DMR for the month in which drilling of the well commenced. Analyses must be conducted by absorption spectrophotometry and results expressed as mg/kg (dry weight) of barite.

2.2.7.6.2 If more than one well is drilled at a site, new analyses are not required for subsequent wells if no new supplies of barite have been received since the previous analysis. In this case, the DMR should state that no new barite was received since the last reported analysis. A permittee may provide certification, as documented by the supplier(s), that the barite meets the above limits. The concentration of mercury and cadmium in stock barite must be reported on the DMR as documented by the supplier.

2.2.8 Environmental Monitoring Requirements

2.2.8.1 All Exploratory Facilities. Monitoring of the fate and effects of drilling muds and/or cuttings discharges are required for all facilities.

2.2.8.2 Environmental Monitoring Study. Operators of all exploratory facilities discharging drilling muds and cuttings must submit a plan of study for environmental monitoring to DEC for review with, or prior to, submission of an NOI.

2.2.8.3 Objectives. The objectives of the environmental monitoring must be to:

2.2.8.3.1 Monitor for discharge related impacts,

2.2.8.3.2 Determine statistically significant changes in sediment pollution concentrations and sediment toxicity with time and distance from the discharge,

- 2.2.8.3.3 Monitor for discharge related impacts to the benthic community,
 - 2.2.8.3.4 Assess whether any impacts warrant an adjustment of the monitoring program, and
 - 2.2.8.3.5 Provide information for permit reissuance.
- 2.2.8.4 Plan of Study. The monitoring must include, but not be limited to, relevant hydrographic, sediment hydrocarbon, and heavy metal data from surveys conducted before and during drilling mud disposal and up to at least one year after drilling operations cease. The monitoring plan must address:
- 2.2.8.4.1 The monitoring objectives,
 - 2.2.8.4.2 Appropriate null and alternative test hypotheses,
 - 2.2.8.4.3 A statistically valid sampling design,
 - 2.2.8.4.4 All monitoring procedures and methods,
 - 2.2.8.4.5 A quality assurance/quality (QA/QC) control program,
 - 2.2.8.4.6 A detailed discussion of how data will be used to meet, test, and evaluate the monitoring objectives, and
 - 2.2.8.4.7 A summary of the results of previous environmental monitoring as they apply to the proposed program plan.
- 2.2.8.5 Reporting Requirements
- 2.2.8.5.1 The permittee must analyze the data and submit a draft report within 180 days following the completion of sample collection. The report must address the environmental monitoring objectives by using appropriate descriptive and analytical methods to test for and to describe any impacts of the effluent on sediment pollutant concentrations, sediment quality, water quality, and the benthic community. The report must include all relevant QA/QC information, including but not limited to instrumentation, laboratory procedures, detection limits/precision requirements of the applied analyses, and sample collection methodology.
 - 2.2.8.5.2 DEC will review the draft report in accordance with the environmental monitoring objectives and evaluate it for compliance with the requirements of the permit. If revisions to the report are required, the permittee must complete them and submit the final report to DEC within two months of DEC's request. The permittee will be required to correct, repeat, or expand environmental monitoring programs which have not fulfilled the requirements of the permit.
- 2.2.8.6 Modification of Monitoring Program. The monitoring program may be modified if DEC determines that the modification is appropriate. The modified program may include changes in sampling stations, sampling times, and parameters.

2.2.8.7 Exemption. DEC may grant a written exemption to this requirement if the permittee can satisfactorily demonstrate that information on the fate and effects of the discharge is available or the discharge will not have significant impacts on the area of biological significance. An exemption to post-drilling monitoring will be granted if no impact was indicated during drilling. An exemption request must be submitted to DEC for review with, or prior to, submission of an NOI.

2.3 Requirements for Deck Drainage (Discharge 002)

2.3.1 Effluent Limitations and Monitoring Requirements. In addition to the restrictions set out in Section 2.1, the permittee must comply with the following effluent limitations and monitoring requirements.

Table 3: Effluent Limitations and Monitoring Requirements Deck Drainage (Discharge 002)

| Parameter | Units | Effluent Limitations | Monitoring Requirements | |
|---|-----------------|----------------------|------------------------------------|---------------------|
| | | | Sample Frequency | Sample Type |
| Free Oil | --- | No Discharge | Daily ¹ | Visual ² |
| Whole Effluent Toxicity (WET) ^{3, 4} | TU _c | Report | Once during first year of coverage | See Section 2.7 |
| Flow | mgd | Report | Monthly | Estimated |
| Footnotes: 1. When discharging. The monitoring frequency is reduced to monthly if the permittee has complied with this requirement for three consecutive months. 2. If discharge occurs during broken or unstable ice conditions or during stable ice conditions, the Static Sheet Test must be used (see 40 CFR Part 435 Subpart A, Appendix 1) and a grab sample is required. 3. Contaminated deck drainage must be processed through an oil-water separator prior to discharge (See Section 2.1.7) and samples for that portion of the deck drainage collected from the separator effluent must be sampled for WET testing. 4. Sample must be collected during a significant rainfall or snow melt. If discharge of deck drainage is initiated after the first year of the permit, sampling must occur during the year following the initiation of separate deck drainage discharge. | | | | |

2.3.2 The permittee must ensure that deck drainage contaminated with oil and grease is processed through an oil-water separator prior to discharge. Once per discharge event, the permittee must sample deck drainage discharges that are processed through the oil-water separator and test for sheen using the Static Sheet Test in 40 CFR Part 435, subpart A.

2.4 Requirements for Domestic Wastewater (Discharge 003)

2.4.1 A permittee using a Marine Sanitation Device (MSD) must conduct annual testing of the MSD to ensure that the unit is operating properly and submit annual test certificates with the December DMR.

2.4.2 Effluent Limitations and Monitoring Requirements. In addition to the restrictions set out in Section 2.1, the permittee must comply with the following effluent limitations and monitoring requirements.

Table 4: Effluent Limitations and Monitoring Requirements for Domestic Wastewater (Discharge 003)

| Discharge | Effluent Parameter | Effluent Limitations | | Monitoring Requirements | |
|--|--------------------|----------------------|-------------------------------|-------------------------|--------------------------|
| | | Avg. Monthly Limit | Max. Daily Limit | Sample Frequency | Sample Type |
| Domestic Wastewater, All Discharges ¹ | Flow Rate (mgd) | Report | | 1/Month | Estimate |
| | TRC | - | 1.0 mg/L Minimum ² | 1/Month | Grab |
| | TRC | - | 1.0 mg/L ³ | 1/Month | Grab |
| | TRC | - | 0.0075 mg/L ⁴ | 1/Month | Grab |
| | Floating Solids | No Discharge | | 1/Day | Observation ⁵ |
| M10 MSD and MSD/BTUs | BOD | 30 mg/l | 60 mg/l | 1/Month | Grab |
| | TSS | 51 mg/l | 67 mg/l | 1/Month | Grab |
| M9IM MSD and MSD/BTUs | BOD | 30 mg/l | 60 mg/l | 1/Month | Grab |
| | TSS | 51 mg/l | 67 mg/l | 1/Month | Grab |
| M10 BTUs | BOD | 30 mg/l | 60 mg/l | 1/Month | Grab |
| | TSS ⁶ | 30 mg/l | 60 mg/l | 1/Month | Grab |
| M9IM BTUs | BOD | 48 mg/l | 90 mg/l | 1/Month | Grab |
| | TSS ⁶ | 56 mg/l | 108 mg/l | 1/Month | Grab |

Footnotes:

1. In cases where domestic wastewater (black water) and graywater are mixed prior to discharge, and sampling of the domestic wastewater stream is infeasible, the discharge may be sampled after mixing. In such cases, the most stringent discharge limits for both discharges shall apply to the mixed stream.
2. Total residual chlorine is a surrogate parameter for fecal coliform and enterococci. For M10 facilities, maintain as close to the minimum limit concentration of 1.0 mg/L as practicable and measure immediately after chlorination. For all other facilities, monitor and report.
3. The 1.0 mg/L maximum daily limit is measured immediately prior to discharge and applies to facilities

authorized a 100-meter mixing zone by DEC.

4. The 0.0075 mg/L limit is measured immediately prior to discharge and applies to permittees not authorized a mixing zone by DEC. The analytical detection limit for this parameter is 0.1 mg/L and will be used as the compliance level for this parameter.
5. The permittee must monitor by observing the surface of the receiving water in the vicinity of the outfall(s) during daylight at the time of maximum estimated discharge and during conditions when observation on the surface of the receiving water is possible in the vicinity of the discharge. For domestic wastewater, observations must follow either the morning or midday meal. Observations must be recorded in daily operating logs and made available upon request by DEC.
6. The TSS limit for BTUs is a net value. For those facilities that use filtered seawater for flushing and treat with BTUs, the TSS of the effluent may be reported as the net value by subtracting the TSS value of the intake water from the TSS value of the effluent. Report the TSS value of the intake water on the comment section of the DMR. Samples collected to determine the TSS value of the intake water must be taken on the same day, and during the same time period that the effluent sample is taken. Intake water samples must be taken at the point where the water enters the facility prior to mixing with other flows. Influent samples must be taken with the same frequency that effluent samples are taken.

2.5 Requirements for Graywater (Discharge 004)

2.5.1 Effluent Limitations and Monitoring Requirements. In addition to the restrictions set out in Section 2.1, the permittee must comply with the following effluent limitations and monitoring requirements.

Table 5: Effluent Limitations and Monitoring Requirements for Graywater (Discharge 004)

| Effluent Characteristic | Units | Sample Location | Sampling Frequency ¹ | Sample Type |
|---|-----------------------------|------------------------------------|---------------------------------|----------------------|
| Total Flow | Gallons per Day (permitted) | Effluent | Daily | Estimate or Measured |
| Biochemical Oxygen Demand (BOD ₅) | mg/L | Influent and Effluent ² | Monthly | Grab or Composite |
| Total Suspended Solids (TSS) | mg/L | Influent and Effluent ² | Monthly | Grab or Composite |
| Floating Solids | Visual | Effluent | Daily | Observation |
| Foam | Visual | Effluent | Daily | Observation |
| Garbage | Visual | Effluent | Daily | Observation |
| Oily Sheen | Visual | Effluent | Daily | Observation |

Footnotes:

1. Samples will be required during periods of operation.
2. Influent and effluent samples must be taken over approximately the same time period. Results are used to confirm that graywater meets primary treatment standards prior to discharge. See 18 AAC 72.990(50)(B) for primary treatment standards.

- 2.5.2 Flow. The permit includes flow monitoring requirements to measure or estimate the effluent discharge flow for each discharge. DEC will use the flow data to determine the amount of contaminants entering the environment and inform future Department decisions during the permit reissuance.
- 2.5.3 BOD₅. The permit requires monthly influent and effluent monitoring during periods of operation.
- 2.5.4 TSS. The permit requires monthly influent and effluent monitoring during periods of operation.
- 2.5.5 Floating Solids, Foam, Garbage, and Oily Sheen. The permit prohibits floating solids, foam, garbage, and oily sheen and requires a visual observation of the receiving water surface at a minimum frequency of once per day. Monitoring of the effluent for floating solids, foam, garbage, and oily sheen is to determine compliance with narrative effluent limits. Observations must be recorded in daily operating logs and made available upon request by DEC.

2.6 Requirements for Miscellaneous Discharges (Discharges 005-013)

- 2.6.1 Effluent Limitations and Monitoring Requirements. In addition to the restrictions set out in Sections 2.1, the discharge of desalination unit wastes (005); blowout preventer fluid (006); boiler blowdown (007); fire control system test water (008); non-contact cooling water (009); uncontaminated ballast water (010); bilge water (011); excess cement slurry (012); and mud, cuttings, and cement at the seafloor (013) must comply with the following effluent limitations and monitoring requirements:

Table 6: Effluent Limitations and Monitoring Requirements for Miscellaneous Discharges 005-013

| Parameter | Effluent Limitations | | Monitoring Requirements | |
|--------------------|---------------------------|---------------------------|-------------------------|-------------|
| | Average Monthly Limit | Maximum Daily Limit | Sample Frequency | Sample Type |
| Flow (mgd) | Report | | Monthly | Estimate |
| Free Oil | No discharge ¹ | No discharge ¹ | Once/Week ¹ | Visual |
| Chemical Additives | See Sections 2.6.3 | | Monthly | Calculation |
| WET ² | See Section 2.6.4 | | Once/Quarter | Grab |

Footnotes:

1. Miscellaneous discharge is limited to those times that a visible sheen observation is possible unless the operator uses the static sheen method which would require a grab sample. Monitoring shall be performed using the visual sheen method on the surface of the receiving water once per week during periods of slack tide when discharging, or by use of the static sheen method at the operator's option. The number of days a sheen is observed must be recorded. For discharges during stable ice, below ice, to unstable ice or broken ice conditions, a water temperature that approximates surface water temperatures after breakup shall be used. Observations must be recorded in daily operating logs and made available upon request by DEC.
2. Applicable to all discharges to which chemical additives have been added, except Discharges 012 (excess cement slurry) and 013 (mud, cuttings, and cement at the seafloor).

2.6.2 Discharge Specific Limitations

- 2.6.2.1 Desalination Unit Waste Water (Discharge 005). In addition to the limitations and monitoring requirements in Section 2.6.1, the permittee must material safety data sheets and an annual inventory of the quantities and rates of chemicals and biocides that are added to desalination unit waste water. Each annual inventory must be assembled for the calendar year and submitted to the DEC Compliance and Enforcement Program by April 1 for the previous calendar year.
- 2.6.2.2 Uncontaminated Ballast Water (Discharge 009). The permittee shall process all uncontaminated ballast water through an oil-water separator prior to discharge
- 2.6.2.3 Bilge Water (Discharge 011). The permittee shall process all bilge water through an oil-water separator prior to discharge.
- 2.6.3 Chemical Additives. The concentration of treatment chemicals in discharged seawater or freshwater shall not exceed the most stringent of the following three constraints. Compliance with these limitations shall be calculated based on the amount of treatment chemical added to the volume of water discharged.
- 2.6.3.1 The maximum concentrations and any other conditions specified in the EPA product registration labeling if the chemical is an EPA registered product.
- 2.6.3.2 The maximum manufacturers recommended concentration.
- 2.6.3.3 The maximum concentration of 500 mg/L
- 2.6.4 WET Monitoring Requirements. WET testing shall be accomplished in accordance with the monitoring requirements in Sections 2.7 and 2.8 of this permit and the WET trigger values (expressed as Chronic Toxic Units (TU_c)) shown in Tables 7 and 8. This requirement applies to any individual, or commingled, miscellaneous discharges greater than 10,000 gallons per day.

Table 7: WET Trigger Levels for Surface Discharges

| Discharge Rate (permitted) | Critical Dilution | Trigger Values (TU _c) |
|----------------------------|-------------------|-----------------------------------|
| 10,000 – 17,000 | 0.24 % | 417 |
| 17,001 – 22,000 | 0.27 % | 370 |
| 22,001 – 27,000 | 0.29 % | 345 |
| 27,001 – 55,000 | 0.36 % | 278 |
| 55,001 – 150,000 | 0.46 % | 217 |
| Greater than 150,000 | 0.62 % | 161 |

Table 8: WET Trigger Levels for Submerged Pipe Discharges

| Discharge Rate (permitted) | Critical Dilution | Trigger Values in TUc |
|----------------------------|-------------------|-----------------------|
| 10,000 – 17,000 | 0.33% | 303 |
| 17,001 – 22,000 | 0.36% | 278 |
| 22,001 – 27,000 | 0.37% | 270 |
| 27,001 – 55,000 | 0.49% | 204 |
| 55,001 – 150,000 | 0.62% | 161 |
| Greater than 150,000 | 0.99% | 101 |

2.7 WET Testing Requirements

This requirement applies to Deck Drainage (002), Desalination Units (005), Blowout Preventer Fluid (006), Boiler Blowdown (007), Fire Control System Test Water (008), Non-contact Cooling Water (009), Uncontaminated Ballast Water (010), and Bilge Water (011).

- 2.7.1 The permittee must conduct tests on grab effluent samples with one vertebrate and two invertebrate species, as follows.
 - 2.7.1.1 Vertebrate (survival and growth): *Atherinops affinis* (topsmelt). In the event that topsmelt is not available, *Menidia beryllina* (inland silverside) may be used as a substitute. The permittee shall document the substitute species in the next DMR.
 - 2.7.1.2 Invertebrate: For larval development tests, the permittee must conduct tests with a bivalve species, *Crassostrea gigas*, (Pacific Oyster) or *Mytilus sp.* (mussel). For fertilization tests, the permittee must use an echinoderm, *Strongylocentrotus purpuratus* (purple sea urchin) or *Dendraster excentricus* (sand dollar). Due to seasonal variability, testing may be performed during reliable spawning periods (e.g. December through February for mussels; June through August for oysters).
- 2.7.2 The permittee must monitor for toxicity once per quarter during discharge activities. If the effluent complies with the toxicity triggers for 12 consecutive months, the permit allows a reduction in toxicity monitoring to once every six months during discharge activities upon written Department approval.
- 2.7.3 Each year, the permittee must rescreen with the three species listed in 2.7.1.1 and 2.7.1.2 and continue to monitor with the most sensitive species. Rescreening must consist of one test conducted at a different time of year from the previous year's test. After screening is completed, monitoring shall be continued at the frequency required in Table 5 or 2.7.2 of this permit.

- 2.7.4 The presence of chronic toxicity must be estimated as specified in *USEPA Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*, Third Edition (EPA-821-R-02-014). For the bivalve species, chronic toxicity must be estimated as specified in *Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Water to West Coast Marine and Estuarine Organisms* (EPA/600/R-95/136).
- 2.7.5 Results must be reported in TU_c , where $TU_c = 100/IC_{25}$. The reported IC_{25} must be the lowest IC_{25} calculated for the applicable survival, growth or fertilization endpoints.
- 2.7.6 A series of at least five dilutions and a control must be tested. The series must include the critical dilution and two dilutions both above and two below the critical dilution. Test series should also be designed to bracket toxicity end points from previous tests to provide meaningful toxicity information during the next permit reissuance.
- 2.7.7 In addition to those quality assurance measures specified in the methodology, the following quality assurance procedures must be followed:
- 2.7.7.1 If organisms are not cultured by the testing laboratory, concurrent testing with reference toxicants must be conducted, unless the test organism supplier provides control chart data from at least the previous five months of reference toxicant testing. Where organisms are cultured by the testing laboratory, monthly reference toxicant testing is sufficient.
 - 2.7.7.2 If either of the reference toxicant tests or the effluent tests does not meet all test acceptability criteria as specified in the test methods manual, then the permittee must re-sample and re-test as soon as possible.
 - 2.7.7.3 Control and dilution water should be receiving water, or salinity adjusted lab water. If the dilution water used is different from the culture water, a second control, using culture water must also be used.
- 2.7.8 Accelerated Testing
- 2.7.8.1 If chronic toxicity is detected above the permit limits or trigger values set forth in Sections 2.6.4, collection and analysis of one additional sample is required within two weeks of receipt of the test results.
 - 2.7.8.2 If chronic toxicity is not detected in the sample required by Sections 2.7.8.1, the permittee must notify DEC in writing of the results within fifteen (15) days of receipt of the results, and must discuss the cause of the exceedance, and the corrective actions that were taken to reduce the toxicity.

- 2.7.8.3 If chronic toxicity is detected in the sample required by Sections 2.7.8.1 then the permittee must conduct four bi-weekly tests over an eight week period. Accelerated testing must be initiated within fifteen (15) days of receiving the sample results required by Sections 2.7.8.1.

2.7.9 Toxicity Reduction Evaluation (TRE) and Toxicity Identification Evaluation (TIE):

- 2.7.9.1 If chronic toxicity limits or triggers are exceeded during accelerated testing, the permittee must initiate a toxicity reduction evaluation (TRE) in accordance with *Generalized Methodology for Conducting Industrial Toxicity Reduction Evaluations* (EPA/600/2-88/070) within two weeks of the receipt of the test results showing an exceedence. At a minimum, the TRE must include:
- 2.7.9.1.1 Further actions to investigate and identify the cause of toxicity;
 - 2.7.9.1.2 Actions the permittee will take to mitigate the impact of the discharge and to prevent the recurrence of toxicity; and
 - 2.7.9.1.3 A schedule for these actions.
- 2.7.9.2 If a TRE is initiated prior to completion of the accelerated testing, the accelerated testing schedule may be terminated, or used as necessary in performing the TRE.
- 2.7.9.3 The permittee may initiate a Toxicity Identification Evaluation (TIE) as part of the TRE process. Any TIE must be performed in accordance with EPA guidance manuals, Toxicity Identification Evaluation; Characterization of Chronically Toxic Effluents, Phase I (EPA/600/6-91/005F), Methods for Aquatic Toxicity Identification Evaluations, Phase II: Toxicity Identification Procedures for Samples Exhibiting Acute and Chronic Toxicity (EPA/600/R-92/080), and Methods for Aquatic Toxicity Identification Evaluations, Phase III: Toxicity Confirmation Procedures for Samples Exhibiting Acute and Chronic Toxicity (EPA-600/R-92/081).
- 2.7.9.4 The permittee may terminate the TRE/TIE process upon submitting satisfactory information demonstrating the toxicity has been identified and/or controlled and obtaining written approval from the Department.

2.7.10 Reporting

- 2.7.10.1 The permittee must report the results of toxicity testing shall be reported on the DMR for the month in which the tests are conducted.
- 2.7.10.2 The permittee must submit the full report shall be submitted by the end of the month following the month in which the DMR is submitted.
- 2.7.10.3 The full report must consist of,
- 2.7.10.3.1 the toxicity test results,

- 2.7.10.3.2 the dates of sample collection and initiation of each toxicity test,
- 2.7.10.3.3 the flow rate at the time of sample collection, and
- 2.7.10.3.4 the results of the effluent sampling for chemical parameters required for the outfalls.

2.7.11 Test results for chronic tests must be reported according to the procedures described in the most recent edition of EPA *Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms*.

2.8 Reporting of Monitoring Requirements

- 2.8.1 Monitoring required in Section 2 shall be summarized each month on the DMR to be provided with the permittee's authorization or a Department-approved equivalent that provides the same information in a similar format.
- 2.8.2 The submitted DMR must be postmarked, faxed, or submitted electronically via email by the 15th day of the following calendar month to DEC at the address in Appendix A – Standard Conditions, Section 1.1.2.
- 2.8.3 DMRs are required to be submitted monthly even if no discharge occurs. The permittee shall mark the DMR as “no discharge” during periods of no discharge. If there is no discharge from an outfall for several consecutive months, these months may be combined on one form.
- 2.8.4 The permittee must sign and certify all DMRs, reports, and other submittals in accordance with signatory requirements Appendix A – Standard Conditions in Section 1.12.
- 2.8.5 With the exception of TRC, the permittee must use EPA-approved methods under 40 CFR Part 136, adopted by reference at 18 AAC 83.010(f), that can achieve a method detection limit (MDL) less than the effluent limit for all effluent monitoring. For a parameter without an effluent limit in this permit, the permittee must use the most sensitive MDL from an EPA-approved analytical test method necessary for compliance monitoring. The permittee must use an EPA-approved test method for TRC monitoring, but in this permit, sample concentrations below the MDL of the EPA-approved method used or 0.1 mg/L, whichever is lower, will be considered the compliance limit.
- 2.8.6 When reporting for a single sample on the DMR, if a value is less than the MDL, the permittee must report “less than [numeric value of MDL],” and if a value is less than a minimum level (ML), the permittee must report “less than [numeric value of ML].”

- 2.8.7 When calculating a monthly average, zero (0) may be assigned for a value less than the MDL, and the numeric value of MDL may be assigned for a value between the MDL and the ML. If the average value is less than the MDL, the permittee must report “less than [numeric value of MDL],” and if the average value is less than the ML, the permittee must report “less than [numeric value of ML].” If a value is equal to or greater than the ML, the permittee must report and use the actual value. The resulting average value must be compared to the compliance level, ML, in assessing compliance.

2.9 Mixing Zone and Modification of Effluent Limits

- 2.9.1 In accordance with State regulations at 18 AAC 70.240, as amended through June 23, 2003, a mixing zone may be authorized as follows:
- 2.9.1.1 Discharge 001 – Drilling Fluids and Drill Cuttings for Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Copper, Iron, Lead, Mercury, Nickel, Selenium, Silver, Thallium, and Zinc,
 - 2.9.1.2 Discharge 003 – Domestic Wastewater for TRC,
 - 2.9.1.3 Discharge 004 – Graywater for TRC,
 - 2.9.1.4 Discharge 005 – Desalination Unit Wastes for chronic WET,
 - 2.9.1.5 Discharge 006 - Blowout Preventer Fluid for chronic WET,
 - 2.9.1.6 Discharge 007 – Boiler Blowdown for chronic WET,
 - 2.9.1.7 Discharge 008 – Fire Control System Test Water for chronic WET,
 - 2.9.1.8 Discharge 009 – Non-contact Cooling Water for chronic WET,
 - 2.9.1.9 Discharge 010 – Uncontaminated Ballast Water for chronic WET,
 - 2.9.1.10 Discharge 011 - Bilge Water for chronic WET, and
 - 2.9.1.11 Discharge 012 – Muds, Cuttings, and Cement at the Seafloor for Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Copper, Iron, Lead, Mercury, Nickel, Selenium, Silver, Thallium, and Zinc
- 2.9.2 The Department will review the NOI information and authorize a standard size cylindrically shaped 100 meter mixing zone for discharges and parameters listed in 2.9.1.
- 2.9.3 The written authorization from the Department will specify authorized discharges and the parameters for which water quality criteria may be exceeded within an authorized mixing zone.
- 2.9.3.1 The Department will authorize a mixing zone if the proposed discharges listed in the NOI are consistent with permit conditions.

- 2.9.3.2 Within an authorized mixing zone, the Department may authorize exceedences of the water quality criteria of 18 AAC 70.020 for Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Chromium, Copper, Iron, Lead, Mercury, Nickel, Selenium, Silver, Thallium, Zinc, TRC, and chronic WET. All water quality criteria must be met at the boundary of the mixing zone.
- 2.9.3.3 If the Department determines that a mixing zone is not appropriate to protect and maintain existing uses of the water body outside of an authorized mixing zone, an permittee may submit additional information to supplement the NOI or may submit an individual permit application Form 1, Form 2C, and Form 2M.

2.10 Best Management Practices Plan

The following BMP Plan applies to all permittees:

- 2.10.1 Through implementation of the BMP plan, the permittee must prevent or minimize the generation and potential for the release of pollutants from the facility to the waters of the United States through normal and ancillary activities.
- 2.10.2 The permittee must develop and implement a BMP Plan which achieves the objectives and the specific requirements listed in Part 2.10.3. Any existing BMP plans may be modified under this section. The BMP Plan shall be ready to implement at the time of submittal of an NOI. The applicant will certify in a cover letter provided with the NOI stating that the BMP Plan is ready to implement when the NOI is submitted.
- 2.10.3 The BMP Plan should be consistent with the general guidance contained in *Guidance Manual for Developing Best Management Practices* (EPA 833-B-93-004, October 1993) or any subsequent revision. The BMP Plan must include, at a minimum, the following items:
 - 2.10.3.1 A description of the number and quantity of pollutants and the toxicity of effluent generated, discharged, or potentially discharged by the permittee must minimize to the extent feasible by managing each waste stream in the most appropriate manner.
 - 2.10.3.2 A description in the BMP Plan, and any Standard Operating Procedures included in the BMP Plan, of how the permittee will ensure proper operation and maintenance of water management and wastewater treatment systems. BMP Plan elements must be developed in accordance with good engineering practices.

- 2.10.3.3 An examination of each facility component or system for waste minimization opportunities and potential for causing a release of significant amounts of pollutants to waters of the United States due to equipment failure, improper operation, or natural phenomena, such as rain or snowfall, etc. The examination must include all normal operations and ancillary activities including material storage areas, storm water, in-plant transfer, material handling and process handling areas, loading or unloading operations, spillage or leaks, sludge and waste disposal, or drainage from raw material storage.
- 2.10.3.4 A statement of BMP policy that includes management commitment to provide the necessary financial, staff, equipment, and training resources to develop and implement the BMP Plan on a continuing basis.
- 2.10.3.5 A description of the structure, functions, and procedures of the BMP Committee. The BMP Plan must establish a BMP Committee chosen by the permittee responsible for developing, implementing, and maintaining the BMP Plan.
- 2.10.3.6 A description of potential pollutant sources.
- 2.10.3.7 An identification and assessment of risks associated with accidental pollutant releases.
- 2.10.3.8 Standard Operating Procedures to achieve the BMP objectives and specific BMPs in Part 2.10.4.
- 2.10.3.9 An assessment of materials compatibility.
- 2.10.3.10 Description of good housekeeping procedures.
- 2.10.3.11 Schedules and procedures for inspections.
- 2.10.3.12 Schedules and procedures for preventative maintenance and repair.
- 2.10.3.13 A description of site security measures and protocols.
- 2.10.3.14 A description of employee training programs.
- 2.10.3.15 A description of record keeping and reporting procedures.
- 2.10.3.16 A description of evaluation procedures for planned modifications to the facility to ensure that the requirements of the BMP Plan are considered as part of the modifications.
- 2.10.3.17 Illustrations depicting the site and facilities including, but not limited to, final constructed site plans, drawings, process flow diagrams, and maps.

2.10.4 Specific BMPs. The BMP Plan must establish specific BMPs or other measures to achieve the objectives under Permit 2.10.3 and which ensure that the following specific requirements are met:

2.10.4.1 Eliminate the introduction of kitchen oils and greases to the graywater system.

When cleaning pots, dishes, pans, etc., the permittee must remove as much food and oil residue as is practicable before rinsing. Alternate waste receptacles or holding tanks must be used for these materials.

2.10.4.2 Prohibit the discharge or placement of any toxic or hazardous material or related residuals into the graywater system (e.g., laundry units, kitchen sinks, dishwashers, drains, sinks, showers, etc.).

2.10.4.3 Prohibit the discharge or placement of unused soaps, detergents, or pharmaceuticals into the graywater system (e.g., laundry units, kitchen sinks, dishwashers, drains, sinks, showers, etc.).

2.10.4.4 Prescribe the use of phosphate free and non-toxic soaps and detergents to be used on-site for all purposes. These soaps and detergents must be free from toxic and bioaccumulative compounds and shall not lead to extreme shifts in receiving water pH.

2.10.4.5 Select chemical cleaning compounds and disinfectants to minimize the addition of nitrogen and phosphorous-based chemical materials to the discharge.

2.10.4.6 Provide minimization plans for biocides, scale inhibitors, and corrosion inhibitors, and other potentially toxic chemicals used at the facility.

2.10.4.7 Mix chemical additives using manufacturer instructions or per EPA product registration labeling, and limit concentrations to 500 mg/L or less.

2.10.5 Annual Certification. The BMP Plan must be reviewed annually by the facility engineering staff and manager, and the BMP Committee.

2.10.5.1 Certified statement that the above reviews have been completed and that the BMP Plan fulfills the requirements set forth in this permit. The statement must be certified by the dated signatures of each BMP Committee member. The statement must be submitted to DEC on or before January 31 of each year of operation under this permit after the initial BMP Plan submittal.

2.10.6 Documentation. The permittee must maintain a copy of the BMP Plan at the facility and make it available to DEC or an authorized representative upon request.

2.10.7 BMP Plan Modification

- 2.10.7.1 The permittee must modify the BMP Plan whenever there is a change in the facility or in the operation of the facility, which materially increases the generation of pollutants or their release or potential release to waters of the United States.
- 2.10.7.2 The permittee must modify the BMP Plan whenever it is found to be ineffective in achieving the general objective of preventing and minimizing the generation and the potential for the release of pollutants from the facility to waters of the United States and/or the specific requirements of Part 2.10.4.
- 2.10.7.3 Any modifications to the BMP Plan must be consistent with the objectives and specific requirements listed in Part 2.10.3. All changes in the BMP Plan must be reported to DEC with the annual certification required under Permit Part 2.10.5.1.

2.11 Removed Substances

Collected screenings, grit, solids, scum, and other facility residuals, or other pollutants removed in the course of treatment or control of water and wastewaters shall be disposed of in a Department-approved manner and method in accordance with 18 AAC 60, such as to prevent any pollution from such materials from entering waters of the United States.

3.0 SPECIAL CONDITIONS

3.1 Quality Assurance Project Plan

- 3.1.1 The permittee must develop a quality assurance project plan (QAPP) for all monitoring required by this permit. The QAPP must be implemented within 120 days of the effective date of this permit.
- 3.1.2 The QAPP must be designed to assist in planning for the collection and analysis of effluent and receiving water samples in support of the permit and in explaining data anomalies when they occur.
- 3.1.3 Throughout all sample collection and analysis activities, the permittee must use the EPA-approved quality assurance/quality control and chain-of-custody procedures described in *Requirements for Quality Assurance Project Plans* (EPA/QA/R-5) and *Guidance for Quality Assurance Project Plans* (EPA/QA/G-5). The QAPP must be prepared in the format which is specified in these documents.
- 3.1.4 The permittee must amend the QAPP as soon as practicable whenever there is a modification in sample collection, sample analysis, or other procedure addressed by the QAPP.
- 3.1.5 Copies of the QAPP must be kept on site and made available to DEC or an authorized representative upon request.

3.2 Corrective Action Plans

Upon violating a permit requirement, the permittee must implement corrective actions as described in this section to come into compliance with the permit as soon as practicable.

3.2.1 Corrective Action Triggers

Conditions requiring corrective actions might be identified through review of DMRs, routine observations by the permittee, or inspections conducted by DEC (or EPA). The following conditions trigger when the permittee must take action to ensure that the condition is rectified and will not be repeated:

- 3.2.1.1 Violation of one or more effluent limits in Table 4 or Table 5 or any other requirement of the permit.
- 3.2.1.2 Inspection or evaluation of the facility by the permittee or DEC (or EPA) results in the discovery that modifications to pollution control measures are necessary to meet the effluent limits.
- 3.2.1.3 The permittee or DEC (or EPA) determines that pollution control measures do not control discharges as necessary to meet WQS.

3.2.1.4 The permittee or DEC (or EPA) determines that pollution control measures do not control discharges as necessary to meet other State regulatory requirements (e.g. minimum treatment per 18 AAC 72).

3.2.1.5 The permittee or DEC (or EPA) determines that pollution control measures or best management practices are not being properly operated and maintained, or are not having the intended effect in minimizing pollutant discharges.

3.2.2 Corrective Action Assessment

The permittee must conduct a corrective action assessment into the nature, cause, and potential options for eliminating these conditions. The findings of the corrective action assessment must be signed and certified per Standard Conditions Section 1.12, Appendix A and retained in the facility recordkeeping documentation. The assessment must include:

3.2.2.1 A description of the conditions(s) discovered (e.g., the inability to meet the effluent limit or minimum treatment requirements of 18 AAC 72), including the date, time, and locations on the facility where it occurred; the types of impacts observed; and the name, title, and signature of the person who identified the problem and of the person who recorded the problem.

3.2.2.2 An explanation of the cause of the conditions(s), if known. If unknown at the time of the assessment, provide an indication of what steps will be taken to determine the cause.

3.2.2.3 A description of the corrective actions to be taken necessary to eliminate the condition(s), and a schedule of activities for completing such actions within the timeframes established in Section 3.2.3.

3.2.2.4 Record of the date(s) and time(s) of the corrective action; a description of the corrective action implemented; and the name, title, and signature of the person recording this information.

3.2.2.5 An indication of the level of correction action required to remedy the trigger condition. The corrective action levels are presented based on increasing level of effort required; minor adjustments, major adjustments, and major renovations.

3.2.2.5.1 Minor adjustments may include changing operational practices, conducting maintenance, or substitute chemicals used in the treatment.

3.2.2.5.2 Major adjustment may include alterations or repairs that do not require the facility to be taken out of service or put into dry dock. These alterations or repairs could include fixing redundant equipment or installing ancillary equipment that could be brought online without interruption.

3.2.2.5.3 Major renovation is one that can only be performed with the system shut down or the facility in dry dock. This may include treatment system renovations or replacements. Major renovations must be accomplished during the next available or scheduled opportunity that would allow for the work.

3.2.3 Corrective Action Schedules

Schedules are established specifically so that noncompliant conditions are not allowed to persist indefinitely and result in additional permit violations. Schedules for corrective actions are established based on level of effort required. The following deadlines apply for eliminating the problem identified in Section 3.2.1 depending on the level of corrective action determined in the assessment in Section 3.2.2:

- 3.2.3.1 Minor Adjustments: As soon as practicable but no later than one month after discovery of the condition for corrective actions that can be accomplished with relatively simple adjustments to control measures, using existing personnel and resources, and not requiring the facility to shut down or be placed in dry dock. If the facility leaves waters subject to this permit, the corrective actions must be completed before recommencing exploration activities in waters subject to this permit.
- 3.2.3.2 Major Adjustments: Within four months of discovering the condition. If more than four months is required to address the condition, the permittee must notify the Department of why additional time is needed and a date when the corrective action is anticipated to be completed. This information must be recorded in the facility records. During the period immediately following the initial violation and before the corrective action has been completed, the permittee must make every effort to reduce potential environmental harm. If the facility leaves and will no longer operate in waters subject to this permit within four months of discovering the condition, corrective actions must be completed prior to recommencing exploration activities in waters subject to this permit.
- 3.2.3.3 Major Renovations: For corrective actions that require large or comprehensive renovations, alterations, or repairs to the facility that can only be achieved while the facility is shutdown or is in dry dock, the permittee must address the underlying cause of the noncompliance at the next available shutdown period or scheduled dry dock. A longer time may be approved by the Department on a case-by-case basis. The permittee must submit acceptable justification for why the corrective action cannot be completed at the next available shutdown or dry dock and establish a date when the correction is will be completed. This information must be recorded in the facility records.

The facility must return to compliance and/or complete necessary renovations or repairs prior to recommencing exploration activities in waters subject to the permit. A permittee that has a facility in dry dock after incurring a violation and does not take corrective action to alleviate the identified condition will be in violation of the corrective actions section of the permit for every occurrence or discharge after recommencing exploration activities in waters subject to this permit (in addition to any original violations prior to going into dry dock).

APPENDIX A STANDARD CONDITIONS

Appendix A

Standard Conditions

APPENDIX B ACRONYMS

Appendix B

Acronyms

The following acronyms are common terms that may be found in an Alaska Pollutant Discharge Elimination System (APDES) permit.

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| 18 AAC 15 | Alaska Administrative Code. Title 18 Environmental Conservation, Chapter 15: Administrative Procedures |
| 18 AAC 60 | Alaska Administrative Code. Title 18 Environmental Conservation, Chapter 60: Solid Waste Management |
| 18 AAC 70 | Alaska Administrative Code. Title 18 Environmental Conservation, Chapter 70: Water Quality Standards |
| 18 AAC 72 | Alaska Administrative Code. Title 18 Environmental Conservation, Chapter 72: Wastewater Disposal |
| 18 AAC 83 | Alaska Administrative Code. Title 18 Environmental Conservation, Chapter 83: Alaska Pollutant Discharge Elimination System |

All chapters of Alaska Administrative Code, Title 18 are available at the Alaska Administrative Code database <http://www.legis.state.ak.us/cgi-bin/folioisa.dll/aac>

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| 40 CFR | Code of Federal Regulations Title 40: Protection of Environment |
| AAC | Alaska Administrative Code |
| ADNR | Alaska Department of Natural Resources |
| APDES | Alaska Pollutant Discharge Elimination System |
| AS | Alaska Statutes |
| AS 46.03 | Alaska Statutes Title 46, Chapter 03: Environmental Conservation. Available at http://www.legis.state.ak.us/default.htm |
| BOD ₅ | Biochemical Oxygen Demand, 5-day |
| BMP | Best Management Practice |
| BPJ | Best Professional Judgment |
| BPT | Best Practicable Control Technology (currently available) |
| CFR | Code of Federal Regulations |
| CWA | Clean Water Act |

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| DEC | Alaska Department of Environmental Conservation |
| DMR | Discharge Monitoring Report |
| EFH | Essential Fish Habitat |
| EPA | U.S. Environmental Protection Agency |
| ESA | Endangered Species Act |
| FC | Fecal Coliform Bacteria |
| mg/L | Milligrams per Liter |
| MZ | Mixing Zone |
| NMFS | National Marine Fisheries Service |
| NOI | Notice of Intent |
| NPDES | National Pollutant Discharge Elimination System |
| ODCE | Ocean Discharge Criteria Evaluation |
| RCRA | Resource Conservation and Recovery Act |
| SU | Standard Units |
| TRC | Total Residual Chlorine |
| TSS | Total Suspended Solids |
| U.S.C. | United States Code |
| USFWS | United States Fish & Wildlife Service |
| WQS | Water Quality Standards |

APPENDIX C DEFINITIONS

Appendix C

Definitions

The following are common definitions of terms associated with APDES permits. Not all the terms listed may appear in a permit. Consult the footnote references for a complete list of terms and definitions.

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| Alaska Pollutant Discharge Elimination System (APDES) ^a | Means the state's program, approved by EPA under 33 U.S.C. 1342(b), for issuing, modifying, revoking and reissuing, terminating, monitoring and enforcing permits and imposing and enforcing pretreatment requirements under 33 U.S.C. 1317, 1328, 1342, and 1345 |
| Annual | Means once per calendar year |
| Average | Means an arithmetic mean obtained by adding quantities and dividing the sum by the number of quantities |
| Average Monthly Discharge Limitation ^a | Means the highest allowable average of "daily discharges" over a calendar month calculated as the sum of all "daily discharges" measured during a calendar month divided by the number of "daily discharges" measured for that month. |
| Best Management Practices (BMPs) ^a | Means schedules of activities, prohibitions of practices, maintenance procedures, and other management practices to prevent or reduce the pollution of waters of the United States. BMPs also include treatment requirements, operating procedures, and practices to control plant site runoff, spillage or leaks, sludge or waste disposal, or drainage from raw material storage areas. |
| Biochemical Oxygen Demand (BOD) ^c | Means the amount, in milligrams per liter, of oxygen used in the biochemical oxidation of organic matter in five days at 20° C |
| Bypass ^a | Means the intentional diversion of waste streams from any portion of a treatment facility |
| Clean Water Act (CWA) ^a | Means the federal law codified at 33 U.S.C. 1251-1387, also referred to as the Federal Water Pollution Control Act or Federal Water Pollution Control Act Amendments of 1972 |
| Coastal | Means any location in or on a water of the United States landward of the inner boundary of the territorial seas. |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

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| Color ^b | Means the condition that results in the visual sensations of hue and intensity as measured after turbidity is removed |
| Commissioner ^a | Means the commissioner of the Alaska Department of Environmental Conservation or the commissioner's designee |
| Composite Samples | Composite samples must consist of at least eight equal volume grab samples. 24 hour composite sample means a combination of at least eight discrete samples of equal volume collected at equal time intervals over a 24-hour period at the same location. A "flow proportional composite" sample means a combination of at least eight discrete samples collected at equal time intervals over a 24-hour period with each sample volume proportioned according to the flow volume. The sample aliquots must be collected and stored in accordance with procedures prescribed in the most recent edition of <i>Standard Methods for the Examination of Water and Wastewater</i> . |
| Contact Recreation ^b | Means activities in which there is direct and intimate contact with water. Contact recreation includes swimming, diving, and water skiing. Contact recreation does not include wading. |
| Criterion ^b | Means a set concentration or limit of a water quality parameter that, when not exceeded, will protect an organism, a population of organisms, a community of organisms, or a prescribed water use with a reasonable degree of safety. A criterion might be a narrative statement instead of a numerical concentration or limit. |
| Daily Discharge ^a | Means the discharge of a pollutant measured during a calendar day or any 24-hour period that reasonably represents the calendar day for the purposes of sampling. For pollutants measured in units of mass, the "daily discharge" is calculated as the total mass of the pollutant discharged over the day. For pollutants with a limitation expressed in other units of measurement, the "daily discharge" is calculated as the average measurement of the pollutant over the day. |
| Department ^a | Means the Alaska Department of Environmental Conservation |
| Design Flow ^a | Means the wastewater flow rate that the plant was designed to handle |
| Director ^a | Means the commissioner or the commissioner's designee assigned to administer the |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

APDES program or a portion of it, unless the context identifies an EPA director

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| Discharge ^a | When used without qualification, discharge means the discharge of a pollutant |
| Discharge of a Pollutant ^a | Means any addition of any pollutant or combination of pollutants to waters of the United States from any point source or to waters of the contiguous zone or the ocean from any point source other than a vessel or other floating craft that is being used as a means of transportation. Discharge includes any addition of pollutants into waters of the United States from surface runoff that is collected or channeled by humans; discharges through pipes, sewers, or other conveyances owned by a state, municipality, or other person that do not lead to a treatment works; discharges through pipes, sewers, or other conveyances leading into privately owned treatment works; and does not include an addition of pollutants by any indirect discharger. |
| Domestic Wastewater ^c | Means waterborne human wastes or graywater derived from dwellings, commercial buildings, institutions, or similar structures. "Domestic wastewater" includes the contents of individual removable containers used to collect and temporarily store human wastes. |
| Effluent ^b | Means the segment of a wastewater stream that follows the final step in a treatment process and precedes discharge of the wastewater stream to the receiving environment |
| Estimated | Means a way to estimate the discharge volume. Approvable estimations include, but are not limited to, the number of persons per day at the facility, volume of potable water produced per day, lift station run time, etc. |
| Excluded area | Means an area not authorized as a receiving water under a permit |
| Fecal Coliform Bacteria (FC) ^b | Bacteria that can ferment lactose at $44.5^{\circ} + 0.2^{\circ}\text{C}$ to produce gas in a multiple tube procedure. Fecal coliform bacteria also means all bacteria that produce blue colonies in a membrane filtration procedure within 24 ± 2 hours of incubation at $44.5^{\circ} + 0.2^{\circ}\text{C}$ in an M-FC broth. |
| Fish ^b | Means any of the group of cold-blooded vertebrates that live in water and have permanent gills for breathing and fins for locomotion |
| Geometric Mean | The geometric mean is the N^{th} root of the product of N. All sample results of zero will use a value of 1 for calculation of the geometric mean. Example geometric mean calculation: |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

$$\sqrt[4]{12 \times 23 \times 34 \times 990} = 55.$$

| | |
|---|---|
| Grab Sample | Means a single instantaneous sample collected at a particular place and time that represents the composition of wastewater only at that time and place |
| Graywater ^b | Means wastewater from a laundry, kitchen, sink, shower, bath, or other domestic source that does not contain excrement, urine, or combined storm water |
| Influent | Means untreated wastewater before it enters the first treatment process of a wastewater treatment works |
| Maximum Daily Discharge Limitation ^a | Means the highest allowable “daily discharge” |
| Mean ^b | Means the average of values obtained over a specified period and, for fecal coliform analysis, is computed as a geometric mean |
| Measured | Means the actual volume of wastewater discharged using appropriate mechanical or electronic equipment to provide a totalized reading. Measure does not provide a recorded measurement of instantaneous rates. |
| Milligrams per Liter (mg/L) ^b | Means the concentration at which one thousandth of a gram (10^{-3} g) is found in a volume of one liter. It is approximately equal to the unit “parts per million (ppm),” formerly of common use. |
| Mixing Zone ^b | Means a volume of water adjacent to a discharge in which wastes discharged mix with the receiving water |
| Month | Means the time period from the 1 st of a calendar month to the last day in the month |
| Monthly Average | Means the average of daily discharges over a monitoring month calculated as the sum of all daily discharges measured during a monitoring month divided by the number of daily discharges measured during that month |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

| | |
|--|--|
| New Facility | Means a facility that has not operated in the area specified in the Notice of intent (NOI) prior to the submission of the NOI. |
| Offshore | Means offshore of the inner boundary of the territorial seas. |
| Open waters | Means ponds, lakes, streams, rivers, and marine waters not covered by ice. |
| Permittee | Means a company, organization, association, entity, or person who is issued a wastewater permit and is responsible for ensuring compliance, monitoring, and reporting as required by the permit |
| pH ^g | Means a measure of the hydrogen ion concentration of water or wastewater; expressed as the negative log of the hydrogen ion concentration in mg/L. A pH of 7 is neutral. A pH less than 7 is acidic, and a pH greater than 7 is basic. |
| Primary Treatment ^c | Means wastewater treatment that: (a) will subsequently discharge wastewater to land or waters that are not waters of the United States and substantially removes all floating and settleable solids; or uses fine screens with 0.04-inch or smaller openings; or (b) will subsequently discharge wastewater to waters of the United States and uses screening, sedimentation, and skimming adequate to remove at least 30 percent of the biochemical oxygen demanding material and of the suspended solids in the treatment works influent; and disinfection, where appropriate. |
| Principal Executive Officer ^a | Means the chief executive officer of the agency or a senior executive officer having responsibility for the overall operations of a principal geographic unit of division of the agency |
| Pollutant ^a | Means dredged spoil, solid waste, incinerator residue, filter backwash, sewage, garbage, sewage sludge, munitions, chemical wastes, biological materials, radioactive materials (except those regulated under 42 U.S.C. 2011), heat, wrecked or discarded equipment, rock, sand, cellar dirt, and industrial, municipal, or agricultural waste discharged into water |
| Receiving WaterBody | Means lakes, bays, sounds, ponds, impounding reservoirs, springs, wells, rivers, streams, creeks, estuaries, marshes, inlets, straits, passages, canals, the Pacific Ocean, Gulf of Alaska, Bering Sea, and Arctic Ocean, in the territorial limits of the state, and all other bodies of surface water, natural or artificial, public or private, inland or coastal, fresh or salt, which are wholly or partially in or bordering the state or under the jurisdiction of the |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

state. (See “Waters of the U.S.” at 18 AAC 83.990(77))

| | |
|--|--|
| Recommencing Facilities | Those facilities that may have let permit coverage lapse but still meet the coverage requirements of the PERMIT. |
| Report | Report results of analysis. |
| Residual Chlorine | Means chlorine remaining in water or wastewater at the end of a specified contact period as combined or free chlorine. |
| Responsible Corporate Officer ^a | Means a president, secretary, treasurer, or vice-president of the corporation in charge of a principal business function or any other person who performs similar policy or decision making functions for the corporation The Responsible Corporate Officer can also be the manager of one or more manufacturing, production, or operating facilities if the requirements of 18 AAC 83.385(a)(1)(B)(i)-(iii) are met. |
| Secondary Recreation ^b | Means activities in which incidental water use can occur. Secondary recreation includes boating, camping, hunting, hiking, wading, and recreational fishing. Secondary contact recreation does not include fish consumption. |
| Severe Property Damage ^a | Means substantial physical damage to property, damage to the treatment facilities which causes them to become inoperable, or substantial and permanent loss of natural resources which can reasonably be expected to occur in the absence of a bypass. Severe property damage does not mean economic loss caused by delays in production. |
| Sheen ^b | Means an iridescent appearance on the water surface |
| Shellfish ^b | Means a species of crustacean, mollusk, or other aquatic invertebrate with a shell or shell-like exoskeleton in any stage of its life cycle |
| Territorial Seas | Means the belt of the seas measured from the line of ordinary low water along that portion of the coast which is in direct contact with the open sea and the line marking the off shore limit of inland waters, and extending off shore a distance of three miles. |
| Total Suspended Solids (TSS) ^g | Means a measure of the filterable solids present in a sample, as determined by the method specified in 40 CFR Part 136 |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

| | |
|---|---|
| Twice per year | Means two time periods during the calendar year: October through April and May through September |
| Upset ^a | Means an exceptional incident in which there is unintentional and temporary noncompliance with technology-based effluent limitations because of factors beyond the reasonable control of the permittee. An upset does not include noncompliance to the extent caused by operational error, improperly designed treatment facilities, inadequate treatment facilities, lack of preventive maintenance, or careless or improper operation. |
| Wastewater Treatment | Means any process to which wastewater is subjected in order to remove or alter its objectionable constituents and make it suitable for subsequent use or acceptable for discharge to the environment |
| Waters of the United States or Waters of the U.S. | Has the meaning given in 18 AAC 83.990(77) |
| Water Recreation ^b | See contact recreation or secondary recreation |
| Water Supply ^b | Means any of the waters of the United States that are designated in 18 AAC 70 to be protected for fresh water or marine water uses. Water supply includes waters used for drinking, culinary, food processing, agricultural, aquacultural, seafood processing, and industrial purposes. Water supply does not necessarily mean that water in a waterbody that is protected as a supply for the uses listed in this paragraph is safe to drink in its natural state. |
| Week | Means the time period of Sunday through Saturday |

a) See 18 AAC 83

b) See 18 AAC 70.990

c) See 18 AAC 72.990

d) See 40 CFR Part 136

e) See EPA Technical Support Document

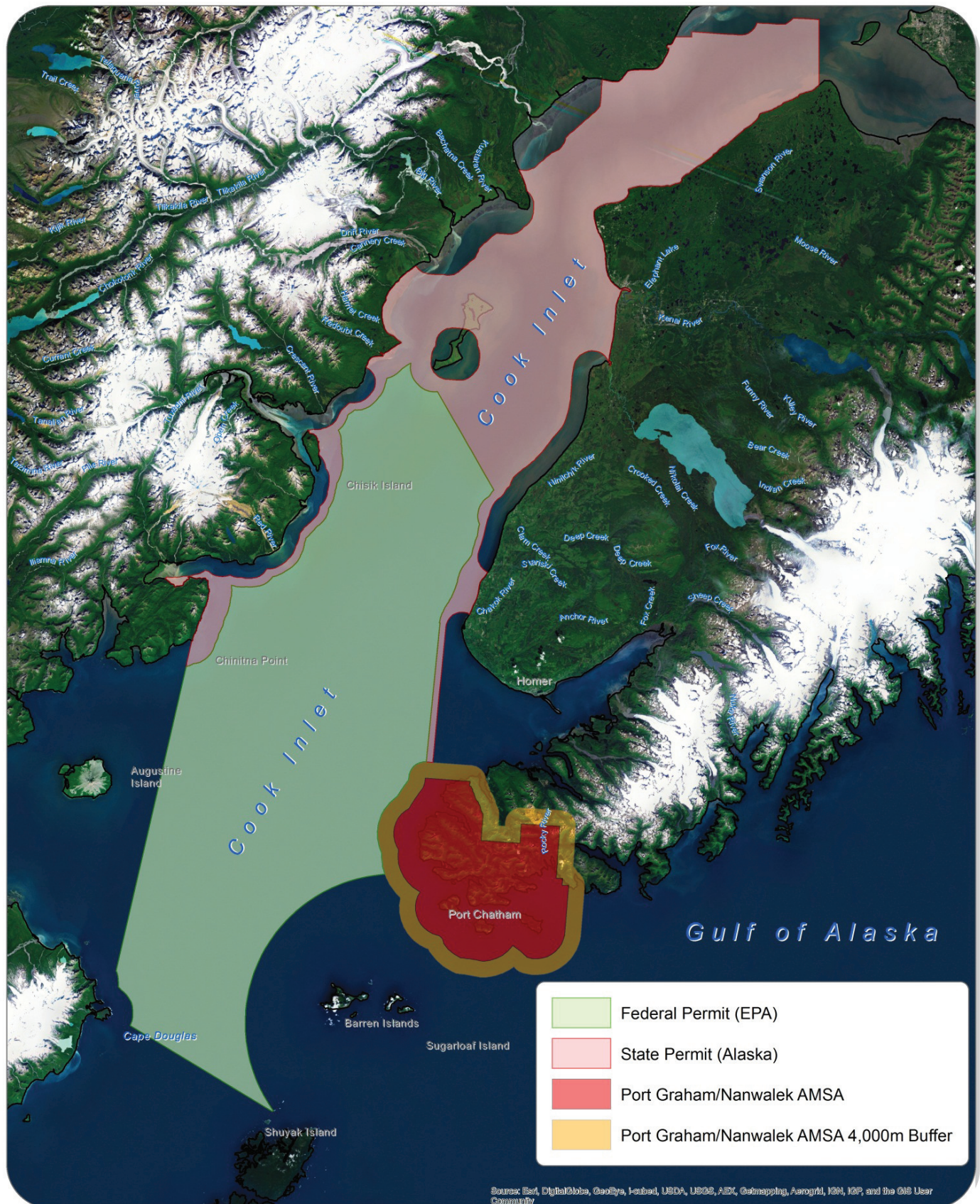
f) See Standard Methods for the Examination of Water and Wastewater 18th Edition

g) See EPA Permit Writers Manual

APPENDIX D COOK INLET COVERAGE AREA

Appendix D

Cook Inlet Coverage Area



Cook Inlet State and Federal Permit Boundaries
with Port Graham AMSA Removed



APPENDIX E SPP METHOD

Appendix E

Method for Conducting a Sediment Toxicity Test with
Leptocherius plumulosus and Non-aqueous Fluids or
Synthetic Based Drilling Muds

Introduction

This test method describes procedures for obtaining data regarding the effects of non-aqueous fluids (NAF) or synthetic based drilling muds (SBMs) on the marine amphipod, *Leptocheirus plumulosus*. The tests are conducted in a similar manner; differences are noted in the text and tables below. USEPA is regulating the sediment toxicity of NAFs and SBMs discharged by oil and gas extraction facilities in coastal and offshore waters as an indication of the toxicity of the drilling muds (USEPA 2000). This test method conforms to the Effluent Limitations Guidelines specified in 40 CFR part 435 (see 66 FR 6849, January 22, 2001). As specified in the Effluent Limitations Guidelines, this test method is consistent with ASTM Standard Guide E 1367-92 (ASTM 1997). Since ASTM E 1367-92 was outdated at the time 40 CFR part 435 (see 66 FR 6849, January 22, 2001) was published in the Federal Register, this test method is also consistent with ASTM E 1367-99 (ASTM 2000), which is the latest version published by ASTM.

Test Species

L. plumulosus is an infaunal amphipod that is indigenous to subtidal regions along the east coast of the U.S. This amphipod constructs U-shaped burrows in the top 5 cm of fine sand to silty clay sediments (ASTM E1367-99). As a result of its broad salinity and particle size tolerances, it is a desirable test species for a variety of toxicity testing programs.

Collection and Handling

In the field, amphipods can be collected using sediment grab samplers such as Peterson and Ponar dredges. This species has been collected in various tributaries of the Chesapeake Bay for various toxicity testing programs (ASTM E 1367-99). The contents of each grab should be sieved through a 500 μ m mesh screen. The sediment and organisms retained on the screen are gently rinsed into plastic buckets containing sediment and water from the collection site. These buckets are quickly transported back to the laboratory and aerated. See ASTM E 1367-99 for more details on collection and handling.

Holding and Acclimation

Amphipods can be placed in aquaria containing a 1-2 cm deep layer of collection site sediment that has been sieved through a 500 μ m mesh screen. Amphipod density should be about 200-300 per 40 L aquarium with vigorous aeration. Two to three days are sufficient for acclimation to test conditions, and during this period a gradual change over from site water to test water is recommended (ASTM E 1367-99).

Environmental Tolerances

L. plumulosus is tolerant of a broad salinity range, from near 0 to 33 g/kg (‰) (ASTM E 1367-99). This species has demonstrated up to 100% survival in >90% silt-clay sediment and an average of 85% survival in >95% sand/gravel sediment (ASTM E 1367-99). The ASTM data are consistent with data published from other studies indicating that *L. plumulosus* is tolerant of sandy and silty sediments. For example, Schlekot et al. (1992) noted a mean survival of 97.5% when *L. plumulosus* was exposed for 10 days to field collected sediments ranging from 98.1% sand to 96.5% fines. Further, this species was collected in

the field in sediments consisting of 99.9% sand and 92.1% fines, indicating that *L. plumulosus* is a generalist and can thrive in a variety of sediment types (Schlekat et al. 1992).

However, the fine fraction of sediments in the Schlekat et al. study did not exceed 55% clay, indicating that the fine fraction was a mixture of silt and clay sized particles. Data from other studies indicated that this species is intolerant of sediments high in clay content. McGee et al. (1999) noted acceptable survival when this species was exposed to Baltimore Harbor sediments containing up to 72% clay. However, Emery et al. (1997) noted significantly reduced amphipod survival when *L. plumulosus* was exposed for 10 days to Magothy River, Maryland sediment (amended with beach sand and kaolinite clay) containing 84%, 90%, and 100% clay.

These data indicated that the tolerance range of this amphipod to clay content is between about 72 to 84%. As such, caution should be used when conducting *L. plumulosus* toxicity tests with sediments with clay content greater than about 70%. This should not have a significant impact on using this species in the NAF and SBM toxicity testing program, since field sediments seldom exceed 70% clay content (Suedel and Rodgers 1991).

Control Sediments

Control sediment must meet certain minimum requirements to be used in the SBM testing program. The primary requirement is that the sediment should be able to support *L. plumulosus* in cultures for extended periods of time. This will ensure that the sediment is chemically nontoxic and that the physical and chemical characteristics of the sediment (e.g., total organic carbon, particle size distribution, and moisture content) are within the tolerance range of the test species. It is expected that separate aliquots of the culture sediment will also be used as a control sediment to be amended by NAFs or SBMs in the NAF/SBM testing program. Any modifications made to the control sediments should be noted in the report.

Characterization

Sediments used in testing should be characterized for total organic carbon (TOC), particle size distribution (sand, silt, and clay), and percent water content. These parameters have been shown to influence the results of NAF/SBM toxicity to *L. plumulosus* in initial experiments. Variations in these sediment characteristics should be quantified so that potential effects of these parameters on test results can be closely monitored.

Collection

Control sediments should be collected from the amphipod collection site or from another area that can provide a consistent source of sediment with characteristics within the tolerance range of *L. plumulosus*. Sediments showing evidence of chemical contamination should not be used in the NAF/SBM testing program. Any site water overlying the sediment should be retained so that fine particles suspended in the water can be re-combined with the sediment before use. Sediment salinity and temperature should be recorded at the time of collection. Sediment collected for use should be homogenized and a composite sample prepared for analysis for the parameters outlined above.

Sieving

Sediments collected in the field for culturing and testing purposes should be first press-sieved through a 2,000 μ m or similar mesh sieve to remove large debris and then through a 500 μ m mesh sieve to remove any indigenous organisms. Sediments have also been press-sieved through a 250 to 350 μ m mesh sieve prior to testing to aid in the enumeration of amphipods on a 500 μ m mesh sieve at test termination.

Storage

The control sediment should be stored in plastic or glass containers at 4 \pm 3 $^{\circ}$ C until test initiation. The sediment should be stored in the dark and should not be allowed to freeze or dry out during storage (E 1367-92).

Test Water

Water used in the NAF/SBM program should be available in sufficient quantities and be acceptable to *L. plumulosus*. The minimum requirement for acceptable water for use in the NAF program is that healthy test organisms survive in the water, and in the water plus control sediment, for the duration of holding and testing without showing signs of disease or stress (ASTM E 1367-99). Another test for acceptability of the test water would be its successful use in the culturing of *L. plumulosus* (with the control sediment).

Natural seawater or synthetic salt water can be used in the NAF program. Natural salt water should be obtained from an uncontaminated area known to support a healthy, reproducing population of *L. plumulosus* or similar sensitive species. Reconstituted salt water can be prepared by adding commercially available sea salt in specified quantities. Natural seawater should be filtered by passing through a 5 micron filter before use. The reader is referred to ASTM E 1367-92 or E 1367-99 for more information concerning test water.

Mixing NAFs or SBMs with Control Sediment

Appendix 3 to Subpart A of Part 435 – Procedure for Mixing Base Fluids with Sediments (40 CFR parts 9 and 435 pages 6901-6902) describes a method for amending control sediments with synthetic-based drilling fluids. This same method can be used to amend control sediments with NAFs and SBMs. The control sediment should be sieved and homogenized before wet to dry weight ratio and density determinations are made and before NAFs are added to the control sediment. The following steps were given in 40 CFR Appendix 3 for mixing NAFs and SBMs with control sediments (parentheses were added here to provide additional information):

1. Determine the wet to dry weight ratio for the control sediment (three replicates of 30 g each has been used successfully);
2. Determine the density (g/ml) of the control sediment (three replicates of >25 ml is suitable for this purpose);
3. Determine the amount of NAF or SBM needed to obtain a desired test concentration;
 - a. Determine the amount of wet sediment required;
 - b. Determine the amount of dry sediment in kilograms for each test concentration;

- c. Determine the amount of NAF or SBM required to amend the control sediment at each test concentration;
4. Mix NAF or SBM with control sediment;
5. Test for homogeneity of NAF or SBM in sediment, and;
6. Mix sufficient quantities of NAF or SBM with control sediment for each treatment of amended or spiked sediment.

The six steps given above for base fluids can also be applied to SBMs, except that the third bullet in Step 3 requires a measurement of the density of the SBM. The density of the SBM can then be used to estimate the quantity required for the desired test concentration. Refer to the formulas below for NAF and SBM calculations:

$$\text{NAF Required (g)} = \frac{\text{Conc Desired (mg/kg)}}{1000\text{g/kg}} \times \frac{\text{Dry Weight Sediment (g)}}{1000 \text{ mg/g}}$$

$$\begin{array}{ccccccc} \text{SBM Required} & = & \text{Conc. Desired} & \times & \text{Dry Weight Sediment} & \times & \text{SBM Density} \\ (\text{g}) & & (\text{ml/kg}) & & (\text{kg}) & & (\text{g/ml}) \end{array}$$

See 40 CFR parts 9 and 435 pages 6901-6902 for more information regarding this procedure.

Mixing Procedure

Mixing the NAF or SBM with the control sediment can be accomplished by following these steps:

- Place appropriate amounts of weighed NAF or SBM into a stainless steel mixing bowl;
- Tare the mixing bowl weight;
- Add appropriate amount of control sediment;
- Mix for 9 to 15 minutes with a hand-held mixer equipped with stainless steel blades (e.g., KitchenAid Model KHM6), and;
- As appropriate, test mixing homogeneity as described below.

The control sediment alone should also be subjected to the mixing procedure to ensure mixing has no effect on sediment toxicity.

Homogeneity of Mixing

As noted above, tests for homogeneity of mixing should be performed, preferably in the procedure development phase (40 CFR part 9 page 6901-6902) by each laboratory performing NAF/SBM toxicity testing. This is to ensure that the NAF or SBM, which can be difficult to homogenize with control sediments, can be evenly mixed with the control sediment by each testing laboratory. Appendix 3 to Subpart A of Part 435 specifies that the coefficient of variation (CV) for a minimum of three replicate samples of the NAF/control sediment mixture must be less than 20%. Determinations of CV should be based on total petroleum hydrocarbon (TPH) content of the NAF or SBM as measured by EPA Methods

3550A and 8015M. If the initial CV is $>20\%$, then the NAF/SBM-sediment mixture must be re-mixed and reanalyzed until the $\leq 20\%$ CV limit is achieved.

Homogeneity measurements should be made on the lowest and highest NAF concentrations for a given test. Laboratories should validate mixing efficiency via TPH measurements (as outlined above) of the low and high NAF concentrations. The homogeneity measurements should be made at least once per year.

Recommended Test Conditions

The recommended test conditions for conducting the 10-day or 96-hr sediment toxicity test with *L. plumulosus* are summarized in Table E-1 and are consistent with methods presented in ASTM E 1367-92 and subsequent updates (E 1367-99). Tests should be conducted at $20 \pm 1^\circ\text{C}$ at $20 \pm 1\%$ salinity with a 14h light; 10 h dark photoperiod at approximately 500-1,000 lux (or about 46 to 93 footcandles). Test chambers are 1-L glass containers with about a 10 cm inside diameter opening (or similar glass containers) that can contain about 150 ml sediment and 600 ml overlying water to achieve a 4:1 (v/v) water to sediment ratio. There are five (5) test concentrations plus a control for each NAF and SBM test. Five (5) replicates are included for the control sediment (E 1367-99) and for each test concentration.

The control sediment/test material mixture and test water should be added to test chambers the day before amphipods are added. This will allow for suspended particles to settle and allow time for equilibration of temperature and the sediment-water interface. After the overnight equilibration period, amphipods are randomly distributed to each test chamber. Twenty amphipods are added to each replicate and there are five replicates per test treatment. Amphipods caught on the water surface can be pushed under with a glass rod. Individuals that have not burrowed within 5 to 10 minutes can be replaced, unless they are exhibiting an avoidance response. Amphipods are not removed at any time during the course of the toxicity test even if they appear dead. Test water is not renewed (i.e., static) and the amphipods are not fed during the exposure period. The toxicity test is terminated after 96 hours or 10 days for SBMs and NAFs respectively.

Temperature, salinity, pH, and dissolved oxygen (DO) should be monitored daily. Ammonia should also be monitored in overlying water to ensure that the concentrations of this constituent do not exceed the tolerance range of the test species. For *L. plumulosus*, this is about 60 mg/L (as total ammonia) at pH 7.7 in 10-day tests (USEPA 1994). Ammonia has not been a problem in initial *L. plumulosus* 96-hr and 10-day tests with various NAFs.

Biological Data

Mortality is the endpoint for *L. plumulosus* at the end of the exposure period. At test termination, the contents of each test chamber (amphipods plus test sediment) are sieved through a 500 um mesh screen to remove amphipods. Material retained on the screen should be rinsed into a sorting tray with clean salt water. The total numbers of live and dead amphipods should be recorded. Missing animals are presumed to have died and decomposed during the test and disintegrated. Amphipods should be counted alive if there are any signs of movement, such as a neuromuscular pleopod twitch (ASTM E 1367-99). Gentle prodding may be used to elicit movement.

Test Acceptability Requirements

Table E-2 provides the acceptability requirements for the 10-day NAF and 96-hr SBM test per ASTM E 1367-92. The primary acceptability requirement for NAF testing is as follows:

- A toxicity test is unacceptable if more than a total of 10% of the control organisms die, or if the coefficient of variation (CV) of control survival is equal to or greater than 40%.

If this acceptability requirement is not met, then the data should be discarded and the experiment repeated. If this requirement is met, then the other acceptability requirements in Table E-2 should be reviewed and a determination made as to the acceptability of the data.

Reference Tests

A single toxicity test will be used to determine satisfactory laboratory performance and to determine whether an NAF or SBM can be discharged as it adheres to drill cuttings. The reference toxicants for the NAF test will be either a C₁₆-C₁₈ -internal olefin reference standard or a C₁₂-C₁₄ or C₈ ester. The reference toxicant for the SBM testing program will be a C₁₆-C₁₈ internal olefin SBM which has also been specified for determining pass/fail for SBMs. The C₁₆-C₁₈ Internal Olefin (IO) SBM is a 65/35 blend, proportioned by mass, of hexadecene and octadecene, respectively (40 CFR part 9 6849). These reference toxicity tests will be conducted in conjunction with all NAF or SBM tests to discern possible changes in the condition of the *L. plumulosus* population used in testing. The reference toxicant test must be conducted concurrently with each sample or batch of samples and at a minimum should be conducted at least monthly. Control charts of this reference standard should be maintained to perform statistical analyses, help understand the inherent variability in the reference test, and for long-term quality control. Test conditions for the reference test should follow the experimental conditions presented in Table E-1.

The reference toxicant test should be performed concurrently and under the same conditions as the NAF or SBM test. The reference toxicant test should be conducted so that control limits (typically set at ± 2 standard deviations) can be established (USEPA 1994). If the reference test LC₅₀ falls outside of this range of control limits generated on the most recent test data points, then the sensitivity of *L. plumulosus* and the credibility of the test results are considered suspect. In this case, the test procedure should be examined and the test repeated with a different batch of amphipods. A sediment test should not automatically be judged unacceptable if the reference test LC₅₀ falls outside the expected range or if the control in the reference toxicity test exceeds 10%. The width of the control limits and all performance criteria listed in Table E-2 should be considered when determining the acceptability of a given NAF or SBM test.

Interpretation of Results

Procedures presented in this test method are used to calculate point estimates, or LC₅₀ values. The LC₅₀ value and 95% confidence limits of the NAF tests should be calculated on the basis of milligrams of NAF per kg dry control sediment (mg/kg) and amphipod mortality. The LC₅₀ value and 95% confidence limits of the NAF tests should be calculated on the basis of milliliters of NAF per kg dry control sediment (ml/kg) and amphipod mortality. A variety of methods can be used to calculate an LC₅₀ value and its 95%

confidence limits, including probit, moving average, trimmed Spearman-Kärber and Litchfield-Wilcoxon methods (ASTM E 1367-99). The method used should take into account the number of partial kills, the number of test chambers per treatment (5), and the number of amphipods per test chamber (20).

The only NAF that will be allowed for use in drilling fluids that are discharge in association with cuttings are those that are as toxic or less toxic, but not more toxic, than the reference NAF (C₁₆-C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester).

The only SBMs that will be allowed for discharge are those that are as toxic or less toxic, but not more toxic, than the C₁₆-C₁₈ internal olefin reference SBM.

These limitations are expressed as follows:

$$\frac{96 \text{ hr. LC}_{50} \text{ RDF}}{96 \text{ hr. LC}_{50} \text{ SBM}} \leq 1.00 \quad \frac{10 \text{ day LC}_{50} \text{ Reference Material}}{10 \text{ day LC}_{50} \text{ NAF}} \leq 1.00$$

Where RDF = Reference Drilling Fluid

The EPA promulgated a sediment toxicity ratio of less than 1.0, indicating that the NAF or SBM can be equally toxic or less toxic, but not more toxic than the reference toxicant test LC₅₀ values for *L. plumulosus*. Hence, the NAF or SBM data should be interpreted by comparing to the reference toxicant test LC₅₀ value and whether it exceeds this value.

Culture Methods

Populations of *L. plumulosus* can be maintained through several generations in the laboratory. The culture conditions specified in ASTM E1367-92 and E1367-99 are provided in Table E-3. Besides the conditions specified, there are other conditions that are important in maintaining healthy *L. plumulosus* cultures, including identifying a source of clean sediment, sieving sediments before use, and the quality of the raw materials used to prepare their food. Preferably, the sediment and water used to culture the amphipods should be collected from the same area as those used in NAF tests. Fine-grained sediments have been shown to be suitable for this purpose (E1367-92). Sediments collected in the field for culturing purposes should be first sieved through a 2,000 μ m mesh sieve to remove large debris and then through a 500 μ m mesh sieve to remove any indigenous organisms. *L. plumulosus* cultures should be maintained at 20 \pm 1 $^{\circ}$ C and 20 \pm 1‰ salinity. If used, natural seawater should be filtered through a 5 micron filter before adding to cultures. New culture chambers should be aerated and allowed to equilibrate overnight before adding amphipods. Water used to start a new culture chamber should be renewed 24 h after initiation and before amphipods are added to culture chambers; otherwise, culture water should be renewed in conjunction with feeding.

Cultures should be observed daily to ensure sufficient aeration. An abundance of amphipods on the sediment surface during daylight hours may indicate insufficient dissolved oxygen or overcrowding, as amphipods typically remain in their burrows unless they are searching for food or a mate. Culture

chambers should be terminated and restarted with fresh sediment about once every 8 weeks to avoid overcrowding. Overcrowding may lead to stress due to food or space limitations, and may also result in reduced female fecundity, thus reducing the relative health of the population of amphipods in a given culture chamber.

Cultures should be routinely inspected for the presence of indigenous worms and copepods, a microbial build-up, or black and sulfurous conditions beneath the sediment surface. Microbial growth appears as a white or gray growth associated with uneaten food, and is indicative of overfeeding. Presence of indigenous species, excess microbial growth, or black and sulfurous conditions may necessitate discarding the affected culture chamber.

Feeding

A mixture of micro-algae, yeast, fish food flakes, alfalfa powder, ground cereal leaves, and shrimp maturation feed has been used to feed cultures (E 1367-92 and E 1367-99). Micro-algae used in culturing include *Pseudoisochrysis paradoxa*, *Phaeodactylum tricornutum*, and *Tetraselmis suecica* mixed in equal parts on a volume basis. These algae provide a source of fatty acids that may otherwise be absent in the diet. In practice, however, it should be noted that *L. plumulosus* has been cultured successfully without the algal mixture and the yeast. The dry food portion of the diet that has been used to successfully culture *L. plumulosus* is shown below.

| Dietary Component | Proportion |
|---|------------|
| Fish food flakes (TetraMin®) | 48.0% |
| Alfalfa powder | 24% |
| Ground cereal leaves (dried wheat leaves) | 24% |
| Shrimp maturation feed (Neo-Novum®) | 4.0% |

This dry food mixture should be homogenized into a fine powder and fed to each culture chamber at a rate of 0.1 to 0.5 g two to three times per week, depending on culture densities. Overfeeding may result in microbial build-up on the sediment surface. The quality of the alfalfa powder and dried wheat leaves may not be consistent among suppliers, thus potentially adversely affecting culture performance. Feeding should occur immediately after culture water changes.

Obtaining Amphipods for Starting a Test

Immature and adult amphipods of mixed sexes and approximately 3 to 5 mm in length (as measured from the base of the first antenna to the end of the third pleon segment along the dorsal surface) are used in toxicity tests, as they are easier to handle and count than younger individuals. Gravid females are not used in testing. The 3 to 5 mm size class individuals are passed through a 1,000 μ m mesh sieve and are retained on a 710 μ m mesh sieve. A 500 μ m mesh sieve has been used previously to retain amphipods of the size needed, but this results in a wider size range of amphipods used for testing. In preliminary NAF

experiments, this wide size range may have contributed to variability in mortality observed that was not present when the 710 μ m mesh sieve was used to retain amphipods in later experiments. The amphipods passing through a 1000 μ m mesh sieve but trapped on a 710 μ m mesh sieve provide a more uniform size range of animals that is thought to decrease the previously-observed variability in mortality. Laboratories are encouraged to use this type of approach to reduce the variability in the size of amphipods used in the NAF/SBM testing program.

Table E- 1: Conditions¹ for conducting 96 hour NAF and 10-day SBM sediment toxicity test with *L. plumulosus*

| Parameter | Conditions |
|--|---|
| Test type | Static whole sediment toxicity test |
| Temperature | 20 \pm 1°C |
| Salinity | 20 \pm 1‰ |
| Light quality | Wide-spectrum fluorescent lights |
| Illuminance | 500-1,000 lux |
| Photoperiod | 14h light:10h dark ² |
| Test chamber | 1-L glass beaker or jar |
| Sediment volume | 150 ml (2 cm depth) |
| Overlying water volume | 600 ml (4:1 [v/v] water to sediment ratio) |
| Renewal of overlying water | None |
| Size and life stage of amphipods | 3-5 mm; immature and adult |
| Number of organisms/chamber | 20 |
| Number of test concentrations | 5 |
| Number of replicate chambers/treatment | 5 in both controls and test treatments |
| Feeding | None |
| Aeration | Water in each test chamber should be aerated throughout the test. |
| Overlying Water | Clean natural or synthetic seawater |
| Overlying water quality | Temperature, salinity, pH, and D.O. daily; ammonia, as needed |
| Test duration | 96 hours |
| Endpoint | Survival |

| | |
|---|---|
| Test acceptability | Minimum mean control survival of 90% and satisfaction of criteria outlined in Table E-2 |
| Dietary Component | Proportion |
| Fish food flakes (TetraMin®) | 48.0% |
| Alfalfa powder | 24% |
| Ground cereal leaves (dried wheat leaves) | 24% |
| Shrimp maturation feed (Neo-Novum®) | 4.0% |
| Footnotes: 1. Conditions listed are consistent with test conditions specified in ASTM E 1367-92 and subsequent updates (E 1367-99) unless otherwise noted 2. Although ASTM E1367 specifies 16h light:8h dark, the photoperiod was changed to 14h light:10h dark to be consistent with the <i>Mysidopsis bahia</i> bioassay for drilling fluids (58 CFR 12453, 1993). | |

Table E- 2: Test acceptability requirements¹ for 10-day NAF and 96 hour SBM test with *L plumulosus*

| |
|---|
| <ul style="list-style-type: none"> • A 10-day NAF and 96-hr SBM toxicity tests are unacceptable if more than a total of 10% of the control organisms die, or if the coefficient of variation (CV) of control survival is equal to or greater than 40%. • Ten-day NAF and 96-hr SBM toxicity tests should usually be considered unacceptable if one or more of the following occurred: • All test chambers were not identical. • Test organisms were not randomly or impartially distributed to test chambers. • Required reference standard was not included in the test. • All test animals were not from the same population, were not all of the same species, or were not of acceptable quality. • Amphipods from a wild population were maintained in the laboratory for more than two weeks, unless the effects of prolonged maintenance in the laboratory has been shown to have no significant effect on sensitivity. • The test organisms were not acclimated at the test temperature and salinity at least 48 hours before they were placed in the test chambers. • Temperature and dissolved oxygen concentrations were not measured. |
|---|

Footnotes:

1. Requirements listed are consistent with those specified in ASTM E 1367-92 and subsequent updates (E 1367-99). However, these guidelines are not identical to those listed ASTM E 1367 in part because some acceptability guidelines listed in E1367-92 are not applicable or practical for the NAF/SBM toxicity testing program

Table E- 3: Culture conditions for *L. plumulosus*

| Parameter | Conditions |
|---|---|
| Temperature | 20±1°C |
| Salinity | 20±1‰ |
| Light quality | Wide-spectrum fluorescent or cool white lights |
| Illuminance | 500-1,000 lux |
| Photoperiod | 14h light:10h dark |
| Culture chamber | Shallow plastic tubs or glass aquaria |
| Sediment volume | 1-2 cm depth at bottom of each culture chamber |
| Renewal of overlying water | Static renewal (30-50% water volume change 2-4 times per week) |
| Number of organisms/chamber | Start with about 300 mixed age (mostly immature and young adults) individuals per chamber |
| Feeding | 0.1 to 0.5 g dry mixture 2-3 times per week (see text) |
| Aeration | Continuous gentle to moderate aeration so as to not suspend sediments |
| Overlying Water | Clean natural or synthetic seawater |
| Overlying water quality | Salinity, temperature, and ammonia during culture start-up |
| Footnotes: | |
| <ol style="list-style-type: none"> 1. Conditions listed are consistent with culture conditions specified in ASTM E 1367-92 and subsequent updates (E 1367-99). | |

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(www.epa.gov/ost/guide/sbf/final/finalfact.html)

**APPENDIX F PROTOCOL FOR THE DETERMINATION OF DEGRADATION OF NON-
AQUEOUS BASED FLUIDS IN A MARINE CLOSED BOTTLE
BIODEGRADATION TEST SYSTEM: MODIFIED ISO 11734**

Appendix F

Protocol for the Determination of Degradation of Non-
aqueous Based Fluids in a Marine Closed Bottle
Biodegradation Test System: Modified ISO 11734

Section 1: Summary of Method

This method determines the anaerobic degradation potential of mineral oils, paraffin oils and non-aqueous fluids (NAF) in sediments. These substrates are base fluids for formulating offshore drilling fluids. The test evaluates base fluid biodegradation rates by monitoring gas production due to microbial degradation of the test fluid in natural marine sediment.

The test procedure places a mixture of marine/estuarine sediment, test substrate (hydrocarbon or controls) and seawater into clean 120 ml (150 ml actual volume) Wheaton serum bottles. The test is run using four replicate serum bottles containing 2000 mg carbon/kg dry weight concentration of test substrate in sediment. The use of resazurin dye solution (1 ppm) evaluates the anaerobic (redox) condition of the bottles (dye is blue when oxygen is present, reddish in low oxygen conditions and colorless if oxygen free). After capping the bottles, a nitrogen sparge removes air in the headspace before incubation begins. During the incubation period, the sample should be kept at a constant temperature of 29 (+/-1)°C. Gas production and composition is measured approximately every two weeks. The samples need to be brought to ambient temperature before making the measurements. Measure gas production using a pressure gauge. Barometric pressure is measured at the time of testing to make necessary volume adjustments.

ISO 11734 specifies that total gas is the standard measure of biodegradation. While modifying this test for evaluating biodegradation of NAF's, methane was also monitored and found to be an acceptable method of evaluating biodegradation. Appendix 1 contains the procedures used to follow biodegradation by methane production. Measurement of either total gas or methane production is permitted. If methane is followed, determine the composition of the gas by using gas chromatography (GC) analysis at each sampling. At the end of the test when gas production stops, or at around 275 days, an analysis of sediment for substrate content is possible. Common methods which have been successfully used for analyzing NAF's from sediments are listed in Appendix 2.

Section 2: System Requirements

This environmental test system has three phases, spiked sediment, overlying seawater, and a gas headspace. The sediment/test compound mixture is combined with synthetic sea water and transferred into 120 mL serum bottles. The total volume of sediment/sea water mixture in the bottles is 75 mL. The volume of the sediment layer will be approximately 50 mL, but the exact volume of the sediment will depend on sediment characteristics (wet:dry ratio and density). The amount of synthetic sea water will be calculated to bring the total volume in the bottles to 75 mL. The test systems are maintained at a temperature of 29 ± 1°C during incubation. The test systems are brought to ambient temperatures prior to measuring pressure or gas volume.

Section 2.1: Sample Requirements

The concentration of base fluids are at least 2000 mg carbon test material/kg dry sediment. Carbon concentration is determined by theoretical composition based on the chemical formula or by chemical analysis by ASTM D5291-96. Sediments with positive, intermediate and negative control substances as well as a C1618 Internal Olefin type base fluid will be run in conjunction with test materials under the same conditions. The positive control is ethyl oleate (CAS 111-62-6), the intermediate control is 1-

hexadecene (CAS 629-73-2), and the negative control is squalane (CAS 111-01-3). Controls must be of analytical grade or the highest grade available. Each test control concentration should be prepared according to the mixing procedure described in Section 3.1.

Product names will be used for examples or clarification in the following text. Any use of trade or product names in this publication is for descriptive use only, and does not constitute endorsement by EPA or the authors.

Section 2.2: Seawater Requirements

Synthetic seawater at a salinity of 25 ± 1 ppt should be used for the test. The synthetic seawater should be prepared by mixing a commercially available artificial seawater mix, into high purity distilled or de-ionized water. The seawater should be aerated and allowed to age for approximately one month prior to use.

Section 2.3: Sediment Requirements

The dilution sediment must be from a natural estuarine or marine environment and be free of the compounds of interest. The collection location, date and time will be documented and reported. The sediment is prepared by press-sieving through a 2000-micron mesh sieve to remove large debris, then press-sieving through a 500-micron sieve to remove indigenous organisms that may confound test results. The water content of the sediment should be less than 60%(w/w) or a wet to dry ratio of 2.5. The sediment should have a minimum organic matter content of 3% (w/w) as determined by ASTM D2974-87 (95) (Method A and D and calculate organic matter as in section 12 of method ASTM D2974-87).

To reduce the osmotic shock to the microorganisms in the sediment the salinity of the sediment's pore water should be between 20-30 ppt. Sediment should be used for testing as soon as possible after field collection. If required, sediment can be stored in the dark at 4°C with 3-6 inches of overlying water in a sealed container for a maximum period of 2 months prior to use.

Section 3: Test Set up

The test is set up by first mixing the test or control substrates into the sediment inoculum, then mixing in seawater to make a pourable slurry. The slurry is then poured into serum bottles, which are then flushed with nitrogen and sealed.

Section 3.1: Mixing Procedure

Because base fluids are strongly hydrophobic and do not readily mix with sediments, care must be taken to ensure base fluids are thoroughly homogenized within the sediment. All concentrations are weight-to-weight comparisons (mg of base fluid to kg of dry control sediment). Sediment and base fluid mixing will be accomplished by using the following method.

- 3.1.1. Determine the wet to dry weight ratio for the control sediment by weighing approximately 10 sub-samples of approximately 1 g each of the screened and homogenized wet sediment into tared aluminum weigh pans. Dry sediment at 105°C for 18-24 h. Remove the dried sediments and cool in a desiccator. Repeat the drying, cooling, and weighing cycle until a

constant weight is achieved (within 4% of previous weight). Re-weigh the samples to determine the dry weight. Calculate the mean wet and dry weights of the 10 sub samples and determine the wet/dry ratio by dividing the mean wet weight by the mean dry weight using Formula 1. This is required to determine the weight of wet sediment needed to prepare the test samples.

$$\frac{\text{Mean Wet Sediment Weight (g)}}{\text{Mean Dry Sediment Weight (g)}} = \text{Wet to Dry Ratio} \quad [1]$$

- 3.1.2. Determine the density (g/ml) of the wet sediment. This will be used to determine total volume of wet sediment needed for the various test treatments. One method is to tare a 5 ml graduated cylinder and add about 5 ml of homogenized sediment. Carefully record the volume then weigh this volume of sediment. Repeat this a total of three times. To determine the wet sediment density, divide the weight by volume per the following formula:

$$\frac{\text{Mean Wet Sediment Weight (g)}}{\text{Mean Dry Sediment Volume (ml)}} = \text{Wet Sediment Density (g/ml)} \quad [2]$$

- 3.1.3. Determine the amount of base fluid to be spiked into wet sediment in order to obtain the desired initial base fluid concentration of 2000 mg carbon/kg dry weight. An amount of wet sediment that is the equivalent of 30 g of dry sediment will be added to each bottle. A typical procedure is to prepare enough sediment for 8 serum bottles (3 bottles to be sacrificed at the start of the test, 4 bottles incubated for headspace analysis, and enough extra sediment for 2 extra bottles). Extra sediment is needed because some of the sediment will remain coated onto the mixing bowl and utensils. Experience with this test may indicate that preparing larger volumes of spiked sediment is a useful practice, then the following calculations should be adjusted accordingly.

- 3.1.3.1 Determine the total weight of dry sediment needed to add 30 g dry sediment to 8 bottles. If more bottles are used then the calculations should be modified accordingly. For example:

$$30 \text{ g dry sediment per bottle} \times 8 = 240 \text{ g dry sediment} \quad [3]$$

- 3.1.3.2 Determine the weight of base fluid, in terms of carbon, needed to obtain a final base fluid concentration of 2000 mg carbon/kg dry weight. For example:

$$\frac{2000 \text{ mg carbon}}{\text{per kg dry sediment}} \times \frac{240 \text{ g}}{1000} = 480 \text{ mg carbon} \quad [4]$$

- 3.1.3.3 Convert from mg of carbon to mg of base fluid.

This calculation will depend on the % fraction of carbon present in the molecular structure of each base fluid. For the control fluids, ethyl oleate is composed of 77.3% carbon, hexadecene is composed of 85.7% carbon, and squalane is composed of 85.3% carbon. The carbon

fraction of each base fluid should be supplied by the manufacturer or determined before use. ASTM D5291-96 or equivalent will be used to determine composition of fluid.

To calculate the amount of base fluid to add to the sediment, divide the amount of carbon (480 mg) by the percent fraction of carbon in the fluid.

For example, the amount of ethyl oleate added to 240 g dry weight sediment can be calculated from the following equation:

$$480 \text{ mg carbon} \div (77.3/100) = 621 \text{ mg ethyl oleate} \quad [5]$$

Therefore, add 621 mg of ethyl oleate to 240 g dry weight sediment for a final concentration of 2000 mg carbon/kg sediment dry weight.

3.1.4. Mix the calculated amount of base fluid with the appropriate weight of wet sediment.

3.1.4.1 Use the wet:dry ratio to convert from g sediment dry weight to g sediment wet weight, as follows:

$$240 \text{ g dry sediment} \times \text{wet:dry ratio} = \text{g wet sediment needed} \quad [6]$$

3.1.4.2 Weigh the appropriate amount of base fluid (calculated in section 3.1.3.3) into stainless mixing bowls, tare the vessel weight, then add the wet sediment calculated in equation 5, and mix with a high shear dispersing impeller for 9 minutes.

The sediment is now mixed with synthetic sea water to form a slurry that will be transferred into the bottles.

Section 3.2: Creating Seawater/Sediment Slurry

Given that the total volume of sediment/sea water slurry in each bottle is to be 75 mL, determine the volume of sea water to add to the wet sediment.

3.2.1 If each bottle is to contain 30 g dry sediment, calculate the weight, and then the volume, of wet sediment to be added to each bottle

$$30 \text{ g dry sediment} \times \text{wet:dry ratio} = \text{g wet sediment added to each bottle} \quad [7]$$

$$\text{g wet sediment} \div \text{density (g/mL) of wet sediment} = \text{mL wet sediment} \quad [8]$$

3.2.2 Calculate volume of sea water to be added to each bottle

$$75 \text{ mL total volume} - \text{mL wet sediment ([8])} = \text{mL of sea water} \quad [9]$$

3.2.3 Determine the ratio of sea water to wet sediment (volume:volume) in each bottle

$$\frac{\text{volume sea water per bottle ([9])}}{\text{volume sediment per bottle ([8])}} = \text{ratio of sea water:wet sediment} \quad [10]$$

3.2.4 Convert the wet sediment weight from equation 6 into a volume using the sediment density.

$$\text{g wet sediment ([6])} \div \text{density} = \text{volume (mL) of sediment} \quad [11]$$

3.2.5 Determine the amount of sea water to mix with the wet sediment.

$$\begin{aligned} \text{mL wet sediment}([11]) \times \text{sea water:sediment ratio}([10]) \\ = \text{mL sea water to add to wet sediment} \quad [12] \end{aligned}$$

Mix sea water thoroughly with wet sediment to form a sediment/sea water slurry.

Section 3.3: Bottling the Sediment Seawater Slurry

The total volume of sediment/sea water slurry in each bottle is to be 75 mL. Convert the volume (mL) of sediment/sea water slurry into a weight (g) using the density of the sediment and the sea water.

3.3.1 Determine the weight of sediment to be added to each bottle

$$\text{mL sediment}([8]) \times \text{density of wet sediment (g/mL)} = \text{g wet sediment} \quad [14]$$

3.3.2 Determine the weight of sea water to be added to each bottle

$$\text{mL sea water}([9]) \times \text{density of sea water (1.01 g/mL)} = \text{g sea water} \quad [15]$$

3.3.3 Determine weight of sediment/sea water slurry to be added to each bottle

$$\text{g wet sediment}([14]) + \text{g sea water}([15]) = \text{g sediment/sea water slurry} \quad [16]$$

This should provide each bottle with 30 g dry sediment in a total volume of 75 mL.

3.3.4 Putting the sediment:seawater slurry in the serum bottles.

Note: The slurry will need to be constantly stirred to keep the sediment suspended.

Place a tared serum bottle on a balance and add the appropriate amount of slurry to the bottle using a funnel. Once the required slurry is in the bottle remove the funnel, add 2-3 drops (25 µl) of a 1gram/L resazurin dye stock solution. Cap the bottle with a butyl rubber stopper (Bellco Glass, Part #2048- 11800)and crimp with an aluminum seal (Bellco Glass Part #2048- 11020).

Using a plastic tube with a (23 gauge, 1 inch long) needle attached to one side and a nitrogen source to the other, puncture the serum cap with the needle. Puncture the serum cap again with a second needle to sparge the bottle's headspace of residual air for two minutes. The nitrogen should be flowing at no more than 100 mL/min to encourage gentle displacement of oxygenated air with nitrogen. Faster nitrogen flow rates would cause mixing and complete oxygen removal would take much longer. Remove the nitrogen needle first to avoid any initial pressure problems. The second (vent) needle should be removed within 30 seconds of removing the nitrogen needle.

Triplicate blank test systems are prepared, with similar quantities of sediment and seawater without any base fluid. Incubate in the dark at a constant temperature of $29 \pm 1^\circ \text{C}$.

Record the test temperature. The test duration is dependent on base fluid performance, but at a maximum should be no more than 275 days. Stop the test after all base fluids have achieved a

plateau of gas production. At termination, base fluid concentrations can be verified in the terminated samples by extraction and GC analysis according to Appendix 2.

Section 4: Concentration Verification Chemical Analyses

Because of the difficulty of homogeneously mixing base fluid with sediment, it is important to demonstrate that the base fluid is evenly mixed within the sediment sea water slurry that was added to each bottle. Of the seven serum bottles set up for each test or control condition, three are randomly selected for concentration verification analyses. These should be immediately placed at 4°C and a sample of sediment from each bottle should be analyzed for base fluid content as soon as possible. The coefficient of variation (CV) for the replicate samples must be less than 20%. The results should show recovery of at least 70% of the spiked base fluid. Use an appropriate analytical procedure described in Appendix 2 to perform the extractions and analyses. If any set of sediments fail the criteria for concentration verification, then the corrective action for that set of sediments is also outlined in Appendix 2.

The nominal concentrations and the measured concentrations from the three bottles selected for concentration verification should be reported for the initial test concentrations. The coefficient of variation (CV) for the replicate samples must be less than 20%. If base fluid content results are not within the 20% CV limit, the test must be stopped and restarted with adequately mixed sediment.

Section 5 Gas monitoring procedures

Biodegradation is measured by total gas as specified in ISO 11734. Methane production can also be tracked and is described in Appendix 1.

Section 5.1 Total Gas monitoring procedures

Bottles should be brought to room temperature before readings are taken. The bottles are observed to confirm that the resazurin has not oxidized to pink or blue. Total gas production in the culture bottles should be measured using a pressure transducer (one source is Biotech International). The pressure readings from test and control cultures are evaluated against a calibration curve created by analyzing the pressure created by known additions of gas to bottles established identically to the culture bottles. Bottles used for the standard curve contain 75 mL of water, and are sealed with the same rubber septa and crimp cap seals used for the bottles containing sediment. After the bottles used in the standard curve have been sealed, a syringe needle inserted through the septa is used to equilibrate the pressure inside the bottles to the outside atmosphere. The syringe needle is removed and known volumes of air are injected into the headspace of the bottles. Pressure readings provide a standard curve relating the volume of gas injected into the bottles and headspace pressure. No less than three points may be used to generate the standard curve. A typical standard curve may use 0, 1, 5, 10, 20 and 40 ml of gas added to the standard curve bottles.

The room temperature and barometric pressure (to two digits) should be recorded at the time of sampling. One option for the barometer is Fisher Part #02-400 or 02-401. Gas production by the sediment is

expressed in terms of the volume (mL) of gas at standard temperature ($0^{\circ}\text{C} = 273^{\circ}\text{K}$) and pressure (1 atm = 30 inches of Hg) using Eqn.17.

$$V_2 = \frac{P_1 \times V_1 \times T_2}{T_1 \times P_2} \quad [17]$$

Where: V_2 = volume of gas production at standard temperature and pressure

P_1 = barometric pressure on day of sampling (inches of Hg)

V_1 = volume of gas measured on day of sampling (mL)

T_2 = standard temperature = 273°K

T_1 = temperature on day of sampling ($^{\circ}\text{C} + 273 = ^{\circ}\text{K}$)

P_2 = standard pressure = 30 inches Hg

An estimation can be made of the total volume of anaerobic gas that will be produced in the bottles. The gas production measured for each base fluid can be expressed as a percent of predicted total anaerobic gas production.

5.1.1. Calculate the total amount of carbon in the form of the base fluid present in each bottle

Each bottle is to contain 30 g dry weight sediment. The base fluid concentration is 2000 mg carbon/kg dry weight sediment. Therefore:

$$2000 \text{ mg carbon/kg sediment} \times (30 \text{ g}/1000) = 60 \text{ mg carbon per bottle} \quad [18]$$

5.1.2. Theory states that anaerobic microorganisms will convert 1 mole of carbon substrate into 1 mole of total anaerobic gas production

Calculate the number of moles of carbon in each bottle.

The molecular weight of carbon is 12 (i.e. 1 mole of carbon = 12 g). Therefore, the number of moles of carbon in each bottle can be calculated.

$$(60 \text{ mg carbon per bottle}/1000) \div 12 \text{ g/mole} = 0.005 \text{ moles carbon} \quad [19]$$

5.1.3. Calculate the predicted volume of anaerobic gas

One mole of gas equals 22.4 L (at standard temperature and pressure), therefore,

$$0.005 \text{ moles} \times 22.4 \text{ L} = 0.112 \text{ L (or 112 mL total gas production)}. \quad [20]$$

Section 5.2 Gas Venting

If the pressure in the serum bottle is too great for the pressure transducer or syringe, some of the excess gas must be wasted. The best method to do this is to vent the excess gas right after measurement. To do this, remove the barrel from a 10-mL syringe and fill it 1/3 full with water. This is then inserted into the bottle through the stopper using a small diameter (high gauge) needle. The excess pressure is allowed to vent through the water until the bubbles stop. This allows equalization of the pressure inside the bottle to

atmospheric without introducing oxygen. The amount of gas vented (which is equal to the volume determined that day) must be kept track of each time the bottles are vented. A simple way to do this in a spreadsheet format is to have a separate column in which cumulative vented gas is tabulated. Each time the volume of gas in the cultures is analyzed, the total gas produced is equal to the gas in the culture at that time plus the total of the vented gas.

To keep track of the methane lost in the venting procedure, multiply the amount of gas vented each time by the corrected % methane determined on that day. The answer gives the volume of methane wasted. This must be added into the cumulative totals similarly to the total gas additions.

Section 6: Test Acceptability and Interpretation

Section 6.1 Test acceptability

At day 275 or when gas production has plateaued, whichever is first, the controls are evaluated to confirm that the test has been performed appropriately. In order for this modification of the closed bottle biodegradation test to be considered acceptable, all the controls must meet the biodegradation levels indicated in Table F-1. The intermediate control hexadecene must produce at least 30% of the theoretical gas production. This level may be reexamined after two years and more data has been generated.

Table F- 1: Test Acceptability Criteria

| Concentration | Percent Biodegradability as a Function of Gas Measurement | | |
|-------------------|---|---------------------------|---------------------------------|
| | Positive control | Squalane negative control | Hexadecene intermediate control |
| 2000 mg carbon/kg | $\geq 60\%$ theoretical | $\leq 5\%$ theoretical | $\geq 30\%$ theoretical |

Section 6.2 Interpretation

In order for a fluid to pass the closed bottle test, the biodegradation of the base fluid as indicated by the total amount of total gas (or methane) generated once gas production has plateaued (or at the end of 275 days, whichever ever is first) must be greater than or equal to the volume of gas (or methane) produced by the reference standard (internal olefin or ester).

The method for evaluating the data to determine whether a fluid has passed the biodegradation test must use the equations:

$$\frac{\% \text{ Theoretical gas production of reference fluid}}{\% \text{ Theoretical gas production of NAF}} \leq 1.0$$

Where: NAF = stock base fluid being tested for compliance

Reference Fluid = C₁₆-C₁₈ internal olefin or C₁₂-C₁₄ or C₈ ester reference fluid

Appendix F, Part I

Section A1 Methane monitoring procedures

The use of total gas production alone may result in an underestimation of the actual metabolism occurring since CO₂ is slightly soluble in water. An acceptable alternative method is to monitor methane production and total gas production. This is easily done using GC analysis. A direct injection of headspace gases can be made into a GC using almost any packed or capillary column with an FID detector. Unless volatile fuels or solvents are present in the test material or the inocula, the only component of the headspace gas that can be detected using an FID detector is methane. The percent methane in the headspace gas is determined by comparing the response of the sample injections to the response from injections of known percent methane standards. The percent methane is corrected for water vapor saturation using Eqn. 8 and then converted to a volume of dry methane using Eqn. 9.

$$\text{Corrected \% CH}_4 = \frac{\% \text{ CH}_4}{1 - \frac{D \times 22.4 \text{ L/mol}}{18 \text{ g/mol} \times 1000}} \quad [\text{A1}]$$

Where: D = the density of water vapor at saturation (g/m³, can be found in CRC Handbook of Chemistry and Physics) for the temperature of sampling.

$$V_{\text{CH}_4} (\text{ml}) = (S + V) \times \frac{(P - P_w)}{(T + 273)} \times \frac{\text{CH}_4}{100} \times \frac{273}{760} \quad [\text{A2}]$$

where: V_{CH₄} (ml) = the volume of methane in the bottle

S = volume of excess gas production (measured with a pressure transducer)

V = volume of the headspace in the culture bottle (total volume - liquid phase)

P = barometric pressure (mm Hg, measured with barometer)

T = temperature (°C)

P_w = vapor pressure of water at T (mm Hg, can be found in CRC Handbook of Chemistry and Physics)

CH₄ = % methane in headspace gas (after correction for water vapor)

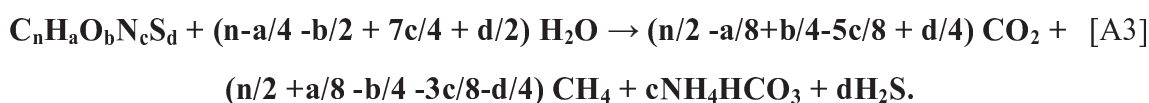
The total volume of serum bottles sold as 125 mL bottles (Wheaton) is 154.8 mL.

The volumes of methane produced are then compared to the volumes of methane in the controls to determine if a significant inhibition of methane production or a significant increase of methane production has been observed. Effective statistical analyses are important, as variability in the results is common due to the heterogeneity of the inoculum's source. It is also common to observe that the timing of the initiation of culture

activity is not equal in all of the cultures. Expect a great variability over the period when the cultures are active, some replicates will start sooner than others, but all of the replicates should eventually reach similar levels of base fluid degradation and methane production.

Section A2 Expected Methane Production Calculations

The amount of methane expected can be calculated using the equation of Symons and Buswell (Eqn. A3). In the case of complete mineralization, all of the carbon will appear as either CO₂ or CH₄, thus the total moles of gas produced will be equal to the total moles of carbon in the parent molecule. The use of the Buswell equation allows you to calculate the effects the redox potential will have on the distribution of the products in methanogenic cultures. More reduced electron donors will allow the production of more methane, while more oxidized electron donors will cause a production of more carbon dioxide.



An example calculation of the expected methane volume in a culture fed 2000 mg/kg hexadecene is as follows. The application of Symons and Buswell's equation reveals that hexadecene (C₁₆H₃₂) will yield 4 moles of CO₂ and 12 moles of CH₄. Assuming 30 g of dry sediment are added to the bottles with 2,334 mg hexadecene/kg dry sediment (i.e. equivalent to 2000 mg carbon/kg dry sediment) the calculation is as follows.

$$\frac{12 \text{ mole CH}_4}{\text{Mole hexadecene}} \times \frac{224 \text{ L}}{\text{mole CH}_4} \times \frac{1000 \text{ ml}}{\text{L}} \times \frac{1 \text{ mole hexadecene}}{2244 \text{ g hexadecene}}$$

$$\times \frac{2.3 \text{ g hexadecane}}{\text{kg dry soil}} \times \frac{0.03 \text{ kg}}{\text{Culture}} = 84 \text{ ml} \quad [\text{A4}]$$

By subtracting the average amount of methane in control bottles from the test bottles and then dividing by the expected volume an evaluation of the completion of the process may be conducted.

Appendix F, Part II

The Concentration Verification analyses is required at the beginning of the test to ensure homogeneity and confirm that the required amount of fluid was delivered to the sediments at the start of the test

- Three samples per fluid need to be analyzed and achieve $\leq 20\%$ Coefficient of Variability and an average of $\geq 70\%$ to $\leq 120\%$ of fluid delivered to sediment.
- If a third party performs the analysis, then the laboratory should be capable of delivering the homogeneity data within seven days, in order to identify any samples that do not meet the homogeneity requirement as quickly as possible.
- If one sediment/fluid set, out a multiple set batch of samples, fails these criteria, then that one set of samples must be discarded and a fresh set of spiked sediment prepared, started, and analyzed to ensure homogeneity. The same stock sediment is used to prepare the replacement set(s). The remaining sets do not need to be re-mixed or restarted.
- The re-mixed set(s) will need to be run the additional days as appropriate to ensure that the total number of days is the same for all sets of bottles, even though the specific days are not aligned.
- Re-mixing of bottle sets can be performed multiple times as a result of a failure of the analytical criteria, until the holding time for the stock sediment has expired (60 days). If the problem set(s) has not fallen within the acceptable analytical criteria by then, it must not be part of the batch of bottles run. If the problem batch is one of the controls, and those controls were not successfully prepared when the sediment holding time expired, then the entire test must be restarted.

References

The following references identify analytical methods that have historically been successful for achieving the analytical quality criteria:

Continental Shelf Associates report 1998. Joint EPA/Industry screening survey to assess the deposition of drill cuttings and associated synthetic based mud on the seabed of the Louisiana continental shelf, Gulf of Mexico. Analysis by Charlie Henry report Number IES/RCAT97-36 GC-FID and GC/MS

EPA Method 3550 for extraction with EPA Method 8015 for GC-FID

Webster, L.; Mackie, P.R.; Hird, S.J.; Munro, P.D.; Brown, N.A. and Moffatt, C.F. (1997) Development of Analytical Methods for the Determination of Synthetic Mud Base Fluids in Marine Sediments *Analyst* 122:1485-1490.

Munro, P.D., B Croce, C.F. Moffet, N.A Brown, A.D. McIntosh, S.J.Hird, R.M. Stagg. 1998. Solid-phase test for comparison for degradation rates of synthetic mud base fluids used in the off shore drilling industry. *Environ. Toxicol. Chem.* 17:1951-1959.

Appendix F, Part III**PROGRAM QUALITY ASSURANCE AND QUALITY CONTROL:****Calibration**

- All equipment / instrumentation will be calibrated in accordance with the test method or the manufacture's instructions and may be scheduled or triggered
- Where possible, standards used in calibration will be traceable to a nationally recognized standard (e.g., certified standard by NIST)
- All calibration activities will be documented and the records retained
- The source, lot, batch number, and expiration date of all reagents used will be documented and retained

Maintenance

- All equipment / instrumentation will be maintained in accordance with the test method or the manufacture's instructions and may be scheduled or triggered
- All maintenance activities will be documented and the records retained

Data Management and Handling

- All primary (raw) data will be correct, complete, without selective reporting, and will be maintained
- Hand-written data will be recorded in lab notebooks or electronically at the time of observation
- All hand-written records will be legible and amenable to reproduction by electrostatic copiers
- All changes to data or other records will be made by:
 - ☐ using a single line to mark-through the erroneous entry (maintaining original data legibility)
 - ☐ write the revision
 - ☐ initial, date, and provide revision code (see attached or laboratory's equivalent)
- All data entry, transcriptions, and calculations will be verified by a qualified person
 - ☐ verification will be documented by initials of verifier and date
- Procedures will be in place to address data management procedures used (at minimum):
 - ☐ Significant figures
 - ☐ Rounding practices
 - ☐ Identification of outliers in data series
 - ☐ Required statistics

Document Control

- All technical procedures, methods, work instructions, standard operating procedures must be documented and approved by laboratory management prior to the implementation

- All primary data will be maintained by the contractor for a minimum of five (5) years

Personnel and Training

- Only qualified personnel shall perform laboratory activities
- Records of staff training and experience will be available. This will include initial and refresher training (as appropriate)

Test Performance

- All testing will done in accordance with the specified test methods
- Receipt, arrival condition, storage conditions, dispersal, and accountability of the test article will be documented and maintained
- Receipt or production, arrival or initial condition, storage conditions, dispersal, and accountability of the test matrix (e.g., sediment or artificial seawater) will be documented and maintained
- Source, receipt, arrival condition, storage conditions, dispersal, and accountability of the test organisms (including inoculum) will be documented and maintained
- Actual concentrations administered at each treatment level will be verified by appropriate methodologies
- Any data originating at a different laboratory will be identified and the laboratory fully referenced in the final report.

**APPENDIX G DETERMINATION OF CRUDE OIL CONTAMINATION IN NON-AQUEOUS
DRILLING FLUIDS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY
(GC/MS)**

Appendix G

Determination of Crude Oil Contamination in Non-aqueous Drilling Fluids by Gas Chromatography/Mass Spectrometry (GC/MS)

1.0 Scope and Application

- 1.1 This method determines crude (formation) oil contamination, or other petroleum oil contamination, in non-aqueous drilling fluids (NAFs) by comparing the gas chromatography/mass spectrometry (GC/MS) fingerprint scan and extracted ion scans of the test sample to that of an uncontaminated sample.
- 1.2 This method can be used for monitoring oil contamination of NAFs or monitoring oil contamination of the base fluid used in the NAF formulations.
- 1.3 Any modification of this method beyond those expressly permitted shall be considered as a major modification subject to application and approval of alternative test procedures.
- 1.4 The gas chromatography/mass spectrometry portions of this method are restricted to use by, or under the supervision of analysts experienced in the use of GC/MS and in the interpretation of gas chromatograms and extracted ion scans. Each laboratory that uses this method must generate acceptable results using the procedures described in Sections 9.2, 10.1, and 13 of this method.

2.0 Summary of Method

- 2.1 Analysis of NAF for crude oil contamination is a step-wise process. Qualitative assessment of the presence or absence of crude oil is performed first. If crude oil is detected in this qualitative assessment, quantitative analysis of the crude oil concentration is performed. When more data are available, the NIST calibration may need to be adjusted.
- 2.2 A sample of NAF is centrifuged, to obtain a solids free supernate.
- 2.3 The sample to be tested is prepared by removing an aliquot of the solids free supernate, spiking it with internal standard, and analyzing it using GC/MS techniques. The components are separated by the gas chromatograph and detected by the mass spectrometer.
- 2.4 Qualitative identification of crude oil contamination is performed by comparing the Total Ion Chromatograph (TIC) scans and Extracted Ion Profile (EIP) scans of test sample to that of uncontaminated base fluids, and examining the profiles for chromatographic signatures diagnostic of oil contamination.
- 2.5 The presence or absence of crude oil contamination observed in the full scan profiles and selected extracted ion profiles determines further sample quantitation and reporting.
- 2.6 If crude oil is detected in the qualitative analysis, quantitative analysis is performed by calibrating the GC/MS using a designated NAF spiked with known concentrations of a designated oil.
- 2.7 Quality is assured through reproducible calibration and testing of GC/MS system and through analysis of quality control samples.

3.0 Definitions

- 3.1 A NAF is one in which the continuous phase is a water immiscible fluid such as an oleaginous material (e.g., mineral oil, enhance mineral oil, paraffinic oil, or synthetic material such as olefins and vegetable esters).
- 3.2 TIC-Total Ion Chromatograph.

3.3 EIP-Extracted Ion Profile.

3.4 TCB-1,3,5-trichlorobenzene is used as the internal standard in this method.

3.5 SPTM-System Performance Test Mix standards are used to establish retention times and monitor detection levels.

4.0 Interferences and Limitations

4.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms.

4.2 All Materials used in the analysis shall be demonstrated to be free from interferences by running method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

4.3 Glassware is cleaned by rinsing with solvent and baking at 400°C for a minimum of 1 hour.

4.4 Interferences may vary from source to source, depending on the diversity of the samples being tested.

4.5 Variations in and additions of base fluids and/or drilling fluid additives (emulsifiers, dispersants, fluid loss control agents, etc.) might also cause interferences and misinterpretation of chromatograms.

4.6 Difference in light crude oils, medium crude oils, and heavy crude oils will result in different responses and thus different interpretation of scans and calculated percentages.

5.0 Safety

5.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however each chemical should be treated as a potential health hazard. Exposure to these chemicals should be reduced to the lowest possible level.

5.2 Unknown samples may contain high concentration of volatile toxic compounds. Sample containers should be opened in a hood and handled with gloves to prevent exposure. In addition, all sample preparation should be conducted in a fume hood to limit the potential exposure to harmful contaminants.

5.3 This method does not address all safety issues associated with its use. The laboratory is responsible for maintaining a safe work environment and a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDSs) should be available to all personnel involved in these analyses. Additional references to laboratory safety can be found in References 16.1 through 16.3.

5.4 NAF base fluids may cause skin irritation, protective gloves are recommended while handling these samples.

6.0 Apparatus and Materials

Note: Brand names, suppliers, and part numbers are for illustrative purposes only. No endorsement is implied. Equivalent performance may be achieved using apparatus and materials other than those specified here, but

demonstration of equivalent performance meeting the requirements of this method is the responsibility of the laboratory.

6.1 Equipment for glassware cleaning.

6.1.1 Laboratory sink with overhead fume hood.

6.1.2 Kiln - Capable of reaching 450°C within 2 hours and holding 450°C within $\pm 10^\circ\text{C}$, with temperature controller and safety switch (Cress Manufacturing Co., Santa Fe Springs, CA B31H or X31TS or equivalent).

6.2 Equipment for sample preparation.

6.2.1 Laboratory fume hood.

6.2.2 Analytical balance - Capable of weighing 0.1 mg.

6.2.3 Glassware.

6.2.3.1 Disposable pipettes - Pasteur, 150 mm long by 5 mm ID (Fisher Scientific 13-678-6A, or equivalent) baked at 400°C for a minimum of 1 hour.

6.2.3.2 Glass volumetric pipettes or gas tight syringes - 1.0-mL $\pm 1\%$ and 0.5-mL $\pm 1\%$.

6.2.3.3 Volumetric flasks - Glass, class A, 10-mL, 50-mL and 100-mL.

6.2.3.4 Sample vials - Glass, 1- to 3-mL (baked at 400°C for a minimum of 1 hour) with PTFE-lined screw or crimp cap.

6.2.3.5 Centrifuge and centrifuge tubes - Centrifuge capable of 10,000 rpm, or better, (International Equipment Co., IEC Centra MP4 or equivalent) and 50-mL centrifuge tubes (Nalgene, Ultratube, Thin Wall 25'89 mm, #3410-2539).

6.3 Gas Chromatograph/Mass Spectrometer (GC/MS):

6.3.1 Gas Chromatograph-An analytical system complete with a temperature-programmable gas chromatograph suitable for split/splitless injection and all required accessories, including syringes, analytical columns, and gases.

6.3.1.1 Column - 30 m (or 60 m) x 0.32 mm ID (or 0.25 mm ID) 1 μm film thickness (or 0.25mm film thickness) silicone-coated fused-silica capillary column (J&W Scientific DB-5 or equivalent).

6.3.2 Mass Spectrometer - Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts (nominal) electron energy in the electron impact ionization mode (Hewlett Packard 5970MS or comparable).

6.3.3 GC/MS interface - the interface is a capillary-direct interface from the GC to the MS.

6.3.4 Data system - A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The

computer must have software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundance versus retention time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EIP). Software must also be available that allows integrating the abundance in any total ion chromatogram (TIC) or EIP between specified retention time or scan-number limits. It is advisable that the most recent version of the EPA/NIST Mass Spectral Library be available.

7.0 Reagents and Standards

- 7.1 Methylene chloride - Pesticide grade or equivalent. Used when necessary for sample dilution.
- 7.2 Standards - Prepare from pure individual standard materials or purchased as certified solutions. If compound purity is 96% or greater, the weight may be used without correction to compute the concentration of the standard.
 - 7.2.1 Crude Oil Reference - NIST 1582 Petroleum Crude Oil Standard Reference Material (U.S. Department of commerce national Institute of Standards and Technology, Gaithersburg, MD 20899) . This oil will be used in the calibration procedures.
 - 7.2.2 Synthetic Base Fluid - Obtain a sample of clean NAF base fluid (as sent from the supplier-has not been circulated downhole). This NAF base fluid will be used in the calibration procedures.
 - 7.2.3 Internal standard - Prepare a 0.01 g/mL solution of 1,3,5-trichlorobenzene (TCB). Dissolve 1.0 g of TCB in methylene chloride and dilute to volume in a 100-mL volumetric flask. Stopper, vortex, and transfer the solution to a 150-mL bottle with PTFE-lined cap. Label appropriately, and store at 5°C to 20°C. Mark the level of the meniscus on the bottle to detect solvent loss.
 - 7.2.4 GC/MS system performance test mix (SPTM) standards - The SPTM standards used in the development of this method contained octane, decane, dodecane, tetradecane, tetradecene, toluene, ethylbenzene, 1,2,4-trimethylbenzene, 1-methylnaphthalene and 1,3-dimethylnaphthalene. These compounds can be purchased individually, obtained as a mixture, or substituted for by a comparable mixture (i.e. Supelco, Catalog No.4-7300). Prepare a high concentration of the SPTM standard at 62.5 mg/mL (total SPTM mixture) in methylene chloride. Prepare a medium concentration SPTM standard at 1.25 mg/mL by transferring 1.0 mL of the 62.5 mg/mL solution into a 50 mL volumetric flask and diluting to the mark with methylene chloride. Finally, prepare a low concentration SPTM standard at 0.125 mg/mL by transferring 1.0 mL of the 1.25 mg/mL solution into a 10-mL volumetric flask and diluting to the mark with methylene chloride.
 - 7.2.5 Crude oil/drilling fluid calibration standards - Prepare a 4-point crude oil/drilling fluid calibration at concentrations of 0% (no spike-clean drilling fluid), 0.5%, 1.0%, and 2.0% by volume according to the procedures outlined below using the Reference Crude Oil:

- 7.2.5.1 Label 4 vials with the following identification: Vial 1-0%Crude in NAF drilling fluid, Vial 2-0.5%Crude in NAF drilling fluid, Vial 3-1%Crude in NAF drilling fluid, and Vial 4-2%Crude in NAF drilling fluid.
 - 7.2.5.2 Vial 1 will not be spiked with Reference Oil in order to retain a “0%” oil concentration, add 5 mL of clean NAF base fluid only.
 - 7.2.5.3 Weigh 90.5 mg of NIST Crude Oil into Vial 2 and add 5 mL of clean NAF base fluid. This will be the 0.5% Crude equivalent in NAF mud standard.
 - 7.2.5.4 Weigh 181 mg of NIST Crude Oil into Vial 3 and add 5 mL of clean NAF base fluid. This will be the 1.0% Crude equivalent in NAF mud standard.
 - 7.2.5.5 Weigh 362 mg in NIST Crude Oil in Vial 4 and add 5 mL clean NAF base fluid. This will be the 2.0% Crude Equivalent in NAF mud standard
 - 7.2.5.6 Thoroughly mix the contents of each of the 4 vial by shaking vigorously.
 - 7.2.5.7 Weigh 0.5 g of the mixture from Vial 1 directly into a tared and appropriately labeled GC straight vial. Spike the 0.5-g supernate with 500 uL of the 0.01 g/mL 1,3,5-trichlorobenzene internal standard solution (see 7.2.3), dilute with methylene chloride, cap with a Teflon lined crimp cap, and vortex for ca. 10 sec.
 - 7.2.5.8 Repeat step 7.2.5.7 except use 0.5 g from Vial 2.
 - 7.2.5.9 Repeat step 7.2.5.7 except use 0.5 g from Vial 3.
 - 7.2.5.10 Repeat step 7.2.5.7 except use 0.5 g from Vial 4.
 - 7.2.5.11 These 4 crude/oil drilling fluid calibration standards are now used for qualitative and quantitative GC/MS analysis.
- 7.2.6 Precision and recovery standard (mid level crude oil/drilling fluid calibration standard)-Prepare a mid point crude oil/drilling fluid calibration using NAF base fluid and Reference Oil at a concentration of 1.0% by volume. Prepare this standard according to the procedures outlined in Section 7.2.5.4. Remove and spike with internal standard, as many 0.5-g aliquots as needed to complete the GC/MS analysis (see Section 11.6- bracketing authentic samples every 12 hours with precision and recovery standard) and the initial demonstration exercise described in Section 9.2.
- 7.2.7 Stability of standards
- 7.2.7.1 When not used, standards are stored in the dark, at -5 to -20°C in screw-capped vials with PTFE-lined lids. A mark is placed on the vial at the level of the solution so that solvent loss by evaporation can be detected. The vial is brought to room temperature prior to use.
 - 7.2.7.2 Solutions used for quantitative purposes shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standard

will remain acceptable if the peak area remains within $\pm 15\%$ of the area obtained in the initial analysis of the standard.

8.0 Sample Collection Preservation and Storage

- 8.1 NAF samples and base fluid samples are collected in 100-to 200-mL glass bottles with PTFE-or aluminum foil lined caps.
- 8.2 Samples collected in the field will be stored refrigerated until time of preparation (not necessary for routine sample).
- 8.3 Sample and extract holding times for this method have not yet been established. However, based on tests experience samples should be analyzed within seven to ten days of collection and extracts analyzed within seven days of preparation.
- 8.4 After completion of GC/MS analysis, extracts should be refrigerated at ca. 4°C until further notification of sample disposal.

9.0 Quality Control

- 9.1 Each laboratory that uses this method is required to operate a formal quality assurance program (Reference 16.4). The minimum requirements of this program consist of an initial demonstration of laboratory capability, and ongoing analysis of standards, and blanks as a test of continued performance, analyses of spiked samples to assess accuracy and analysis of duplicates to assess precision. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.
 - 9.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 9.2.
 - 9.1.2 The analyst is permitted to modify this method to improve separations or lower the cost of measurements, provided all performance requirements are met. Each time a modification is made to the method, the analyst is required to repeat the calibration (Section 10.4) and to repeat the initial demonstration procedure described in Section 9.2.
 - 9.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 9.3.
 - 9.1.4 An analysis of a matrix spike sample is required to demonstrate method accuracy. The procedure and QC criteria for spiking are described in Section 9.4.
 - 9.1.5 Analysis of a duplicate field sample is required to demonstrate method precision. The procedure and QC criteria for duplicates are described in Section 9.5.
 - 9.1.6 Analysis of a sample of the clean NAF(s) (as sent from the supplier-has not been circulated downhole) used in the drilling operations is required.

- 9.1.7 The laboratory shall, on an ongoing basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (Section 7.2.6) that the analysis system is in control. These procedures are described in Section 11.6.
- 9.1.8 The laboratory shall maintain records to define the quality of data that is generated.
- 9.2 Initial precision and accuracy-The initial precision and recovery test is performed using the precision and recovery standard (1% by volume Crude Equivalent in NAF drilling fluid). The laboratory shall generate acceptable precision and recovery by performing the following operations.
- 9.2.1 Prepare four separate aliquots of the precision and recovery standard using the procedure outlined in Section 7.2.6. Analyze these aliquots using the procedures outlined in Section 11.
- 9.2.2 Using the results of the set of four analyses, compute the average recovery (\bar{X}) in weight percent and the standard deviation of the recovery (s) for each sample.
- 9.2.3 If s and \bar{X} meet the acceptance criteria of 80% to 110%, system performance is acceptable and analysis of samples may begin. If, however, s exceeds the precision limit or \bar{X} falls outside the range for accuracy, system performance is unacceptable. In this event, review this method, correct the problem, and repeat the test.
- 9.2.4 Accuracy and precision-The average percent recovery (P) and the standard deviation of the percent recovery (Sp) Express the accuracy assessment as a percent recovery interval from $P-2Sp$ to $P+2Sp$. For example, if $P=90\%$ and $Sp=10\%$ for four analyses of crude oil in NAF, the accuracy interval is expressed as 70% to 110%. Update the accuracy assessment on a regular basis.
- 9.3 Blanks-Rinse glassware and centrifuge tubes used in the method with ca. 30 mL of methylene chloride, remove a 0.5-g aliquot of the solvent, spike it with the 500 mL of the internal standard solution (Section 7.2.3) and analyze a 1-mL aliquot of the blank sample using the procedure in Section 11. Compute results per Section 12.
- 9.4 Matrix spike sample-Prepare a matrix spike sample according to procedure outlined in Section 7.2.6. Analyze the sample and calculate the concentration (% oil) in the drilling fluid and % recovery of oil from the spiked drilling fluid using the methods described in Sections 11 and 12.
- 9.5 Duplicates-A duplicate field sample is prepared according to procedures outlined in Section 7.3 and analyzed according to Section 11. The relative percent difference (RPD) of the calculated concentrations should be less than 15%.
- 9.5.1 Analyze each of the duplicates per the procedure in Section 11 and compute the results per Section 12.
- 9.5.2 Calculate the relative percent difference (RPD) between the two results per the following equation:

$$RPD = \frac{D1 - D2}{\quad}$$

$$(D1 + D2)/2$$

where: D1 = Concentration of crude oil in the sample

D2 = Concentration of crude oil in the duplicate sample

- 9.5.3 If the RPD criteria are not met, the analytical system shall be judged to be out of control, and the problem must be immediately identified and corrected and the sample batch re-analyzed.
- 9.6 Preparation of the clean NAF sample is performed according to procedures outlined in Section 7.3 except that the clean NAF (drilling fluid that has not been circulated downhole) is used. Ultimately the oil-equivalent concentration from the TIC or EIP signal measured in the clean NAF sample will be subtracted from the corresponding authentic field samples in order to calculate the true contaminant concentration (% oil) in the field samples (see Section 12).
- 9.7 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for initial precision and recovery (Section 9.2) and ongoing precision and recovery (Section 11.6) shall be identical, so that the most precise results will be obtained. The GC/MS instrument will provide the most reproducible results if dedicated to the setting and conditions required for the analyses given in this method.
- 9.8 Depending on specific program requirements, field replicates and field spikes of crude oil into samples may be required when this method is used to assess the precision and accuracy of the sampling and sample transporting techniques.

10.0 Calibration

- 10.1 Establish gas chromatographic/mass spectrometer operating conditions given in Table G-1 below. Perform the GC/MS system hardware-tune as outlined by the manufacture. The gas chromatograph is calibrated using the internal standard technique. Note: Because each GC is slightly different, it may be necessary to adjust the operating conditions (carrier gas flow rate and column temperature and temperature program) slightly until the retention times in Table G-2 are met.

Table G- 1: Gas Chromatograph/Mass Spectrometer (GS/MS) Operating Conditions

| Parameter | Setting |
|---------------------|---|
| Injection port | 280°C |
| Transfer line | 280°C |
| Detector | 280°C |
| Initial Temperature | 50°C |
| Initial Time | 5 minutes |
| Ramp | 50 to 300°C@ 5°C per minute |
| Final Temperature | 300°C |
| Final Hold | 20 minutes or until all peaks have eluted |
| Carrier Gas | Helium |
| Flow rate | As required for standard operation |
| Split ratio | As required to meet performance criteria (~1:100) |
| Mass range | 35 to 600 amu |

Table G- 2: Approximate Retention Times for Compounds

| Compound | Approximate Retention Time (minutes) |
|--------------------------|--------------------------------------|
| Toulene | 5.6 |
| Octane, n-C8 | 7.2 |
| Ethylbenzene | 10.3 |
| 1,2,4 – Trimethylbenzene | 16.0 |
| Decane, n-C10 | 16.1 |
| TCB (Interal Standard) | 21.3 |
| Dodecane, n-C12 | 22.9 |
| 1-Methylnaphthalene | 26.7 |
| 1-Tetradecene | 28.4 |
| Tetradecane, n-C14 | 28.7 |
| 1,3-Dimethylnaphthalene | 29.7 |

10.2 Internal standard calibration procedure-1,3,5-trichlorobenzene (TCB) has been shown to be free of interferences from diesel and crude oils and is a suitable internal standard.

10.3 The system performance test mix standards prepared in Section 7.2.4 are primarily used to establish retention times and establish qualitative detection limits.

- 10.3.1 Spike a 500 uL aliquot of the 1.25 mg/mL SPTM standard with 500 uL of the TCB internal standard solution.
- 10.3.2 Inject 1.0 uL of this spiked SPTM standard onto the GC/MS in order to demonstrate proper retention times. For the GC/MS used in the development of this method the ten compounds in the mixture had typical retention times shown in Table G-2 above. Extracted ion scans for m/z 91 and 105 showed a maximum abundance of 400,000.
- 10.3.3 Spike a 500 uL aliquot of the 0.125 mg/mL SPTM standard with 500 uL of the TCB internal standard solution.
- 10.3.4 Inject 1.0 mL of this spiked SPTM standard onto the GC/MS to monitor detectable levels. For the GC/MS used in the development of this test all ten compounds showed a minimum peak height of three times signal to noise. Extracted ion scans for m/z 91 and 105 showed a maximum abundance of 40,000.

10.4 GC/MS crude oil/drilling fluid calibration -There are two methods of quantification: Total Area Integration (C8-C13) and EIP Area Integration using m/z's 91 and 105. The EIP Area Integration method should be used as the primary method for quantifying oil in NAFs and enhanced mineral oil (EMO) based drilling fluid. Inject 1.0 μ L of each of the four crude oil/drilling fluid calibration standards prepared in Section 7.2.5 into the GC/MS. The internal standard should elute approximately 21-22 minutes after injection. For the GC/MS used in the development of this method, the internal standard peak was (35 to 40)% of full scale at an abundance of about 3.5×10^7 .

- 10.4.1 Total Area Integration Method-For each of the four calibration standards obtain the following: Using a straight baseline integration technique, obtain the total ion chromatogram (TIC) area from C8 to C13. Obtain the TIC area of the internal standard (TCB). Subtract the TCB area from the C8-C13 area to obtain the true C8-C13 area. Using the C8-C13 and TCB areas, and known internal standard concentration, generate a linear regression calibration using the internal standard method. The r^2 value for the linear regression curve should be ≥ 0.998 . Some synthetic fluids might have peaks that elute in the window and would interfere with the analysis. In this case the integration window can be shifted to other areas of scan where there are no interfering peaks from the synthetic base fluid.
- 10.4.2 EIP Area Integration-For each of the four calibration standards generate Extracted Ion Profiles (EIPs) for m/z 91 and 105. Using straight baseline integration techniques, obtain the following EIP areas:

- 10.4.2.1 For m/z 91 integrate the area under the curve from approximately 10.5 minutes to 25 minutes, including the internal standard. The internal standard area is used in the calculations.
- 10.4.2.2 For m/z 105 integrate the area under the curve from approximately 10.5 minutes to 25 minutes.
- 10.4.2.3 Using the EIP areas for TCB, m/z 91 and m/z 105, and the known concentration of internal standard. Calculate the ratio of the total m/z 105 area divided by the internal standard area at m/z 91. Generate linear regression calibration curves for the ratios using the internal standard method. The r^2 value for each of the EIP linear regression curves should be ≥ 0.998 .
- 10.4.2.4 Some base fluids might produce a background level that would show up on the extracted ion profiles, but there should not be any real peaks (signal to noise ratio of 1:3) from the clean base fluids.

11.0 Procedure

11.1 Sample Preparation-

- 11.1.1 Mix the authentic field sample (drilling fluid) well. Transfer (weigh) a 30-g aliquot of the sample to a labeled centrifuge tube.
- 11.1.2 Centrifuge the aliquot for a minimum of 15 min at approximately 15,000 rpm, in order to obtain a solids free supernate.
- 11.1.3 Weigh 0.5 g of the supernate directly into a tared and appropriately labeled GC straight vial.
- 11.1.4 Spike the 0.5-g supernate with 500 μ L of the 0.01g/mL 1,3,5-trichlorobenzene internal standard solution (see 7.2.3), dilute with methylene chloride, cap with a Teflon lined crimp cap, and vortex for ca. 10 sec.
- 11.1.5 The sample is ready for GC/MS analysis.

11.2 Gas Chromatography. Table G-1 summarizes the recommended operating conditions for the GC/MS. Retention times for the n-alkanes obtained under these conditions are given in Table G-2. Other columns, chromatographic conditions, or detectors may be used if initial precision and accuracy requirements (Section 9.2) are met. The system is calibrated according to the procedures outlined in Section 10, and verified every 12 hours according to Section 11.6.

- 11.2.1 Samples should be prepared (extracted) in a batch of no more than 20 samples. The batch should consist of 20 authentic samples, 1 blank (Section 9.3), 1 matrix spike sample (9.4), and 1 duplicate field sample (9.5), and a prepared sample of the corresponding clean NAF used in the drilling process.
- 11.2.2 An analytical sequence is run on the GC/MS where the 3 SPTM standards (Section 7.2.4) containing internal standard are analyzed first, followed by analysis of the four GC/MS crude

oil/drilling fluid calibration standards (Section 7.2.5), analysis of the blank, matrix spike sample, the duplicate sample, the clean NAF sample, followed by the authentic samples.

- 11.2.3 Samples requiring dilution due to excessive signal should be diluted using methylene chloride.
- 11.2.4 Inject 1.0 uL of the test sample or standard into the GC, using the conditions in Table G-1.
- 11.2.5 Begin data collection and the temperature program at the time of injection.
- 11.2.6 Obtain a TIC and EIP fingerprint scans of the sample (Table G-3).

Table G- 3: Recommended Ion Mass Numbers

| Selected Ion Mass Numbers | Corresponding Aromatic Compounds | Typical Retention Times (minutes) |
|------------------------------|-------------------------------------|--------------------------------------|
| 91 | Methylbenzene | 6.0 |
| | Ethylbenzene | 10.3 |
| | 1,4-Dimethylbenzene | 10.9 |
| | 1,3-Dimethylbenzene | 10.9 |
| | 1,2-Dimethylbenzene | 10.9 |
| 105 | 1,3,5-Trimethylbenzene | 15.1 |
| | 1,2,4-Trimethylbenzene | 16.0 |
| | 1,2,3-Trimethylbenzene | 17.4 |
| 156 | 2,6-Dimethylnaphthalene | 28.9 |
| | 1,2-Dimethylnaphthalene | 29.4 |
| | 1,3-Dimethylnaphthalene | 29.7 |

- 11.2.7 If the area of the C8 to C13 peaks exceeds the calibration range of the system, dilute a fresh aliquot of the test sample weighing < 0.50-g and reanalyze.
- 11.2.8 Determine the C8 to C13 TIC area, the TCB internal standard area, and the areas for the m/z 91 and 105 EIPs. These are used in the calculation of oil concentration in the samples (see Section 12).
- 11.2.9 Observe the presence of peaks in the EIPs that would confirm the presence of any target aromatic compounds. Using the EIP areas and EIP linear regression calibrations determine the amount of crude oil contamination equivalent in the sample.

11.3 Qualitative Identification

- 11.3.1 Qualitative identification is accomplished by comparison of the TIC and EIP area data from an authentic sample to the TIC and EIP area data from the calibration standards (Section 10.4). Crude oil is identified by the presence of C10 to C13 n-alkanes and corresponding target aromatics.
- 11.3.2 Using the calibration data, establish the identity of the C8 to C13 peaks in the chromatogram of the sample. Using the calibration data, establish the identity of any target aromatics present on the extracted ion scans.
- 11.3.3 Crude oil is not present in a detectable amount in the sample if there are no target aromatics seen on the extracted ion scans. The experience of the analyst shall weigh heavily in the determination of the presence of peaks at a signal-to-noise ratio of 3 or greater.
- 11.3.4 If the chromatogram shows n-alkanes from C8 to C13 and target aromatics to be present, contamination by crude oil or diesel should be suspected and quantitative analysis should be determined. If there are no n-alkanes present that are not seen on the blank, and no target aromatics are seen, the sample can be considered to be free of contamination.

11.4 Quantitative Identification

- 11.4.1 Determine the area of the peaks from C8 to C13 as outlined in the calibration section (10.4.1). If the area of the peaks for the sample is greater than that for the clean NAF (base fluid) use the crude oil/drilling fluid calibration TIC linear regression curve to determine approximate crude oil contamination. (This step will be difficult for NAF samples that have measurable amounts of C8 to C13 peaks in the clean fluid. The EIPs should be used for quantitation of crude oil).
- 11.4.2 Using the EIPs outlined in Section 10.4.2 determine the presence of any target aromatics. Using the integration techniques outlined in Section 10.4.2 to obtain the EIP areas for m/z 91 and 105. Use the crude oil/drilling fluid calibration EIP ratio linear regression curves to determine approximate crude oil contamination.

11.5 Complex Samples

- 11.5.1 The most common interferences in the determination of crude oil can be from mineral oil, diesel oil, and proprietary additives in drilling fluids.
- 11.5.2 Mineral oil can typically be identified by its lower target aromatic content, and narrow range of strong peaks.
- 11.5.3 Diesel oil can typically be identified by low amounts of n-alkanes from C7 to C9, and the absence of n-alkanes greater than C25.
- 11.5.4 Crude oils can usually be distinguished by the presence of high aromatics, increased intensities of C8 to C13 peaks, and/or the presence of higher hydrocarbons of C25 and greater (which may be difficult to see in some synthetic fluids at low contamination levels).
 - 11.5.4.1 Oil condensates from gas wells are low in molecular weight and will normally produce strong chromatographic peaks in the C8-C13 range. If a sample of the gas

condensate crude oil from the formation is available, the oil can be distinguished from other potential sources of contamination by using it to prepare a calibration standard.

- 11.5.4.2 Asphaltene crude oils with API gravity <20 may not produce chromatographic peaks strong enough to show contamination at levels of the calibration. Extracted ion peaks should be easier to see than increased intensities for the C8 to C13 peaks. If a sample of asphaltene crude from the formation is available, a calibration standard should be prepared.

11.6 System and Laboratory Performance

- 11.6.1 At the beginning of each 8-hour shift during which analyses are performed, GC crude oil/drilling fluid calibration and system performance test mixes are verified. For these tests, analysis of the medium-level calibration standard (1-% Reference Oil in IO Lab drilling fluid, and 1.25 mg/mL SPTM with internal standard) shall be used to verify all performance criteria. Adjustments and/or re-calibration (per Section 10) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples and blanks be analyzed.
- 11.6.2 Inject 1.0 mL of the medium-level GC/MS crude oil/drilling fluid calibration standard into the GC instrument according to the procedures in Section 11.2. Verify that the linear regression curves for both TIC area and EIP areas are still valid using this continuing calibration standard.
- 11.6.3 After this analysis is complete, inject 1.0 mL of the 1.25 mg/mL SPTM (containing internal standard) into the GC instrument and verify the proper retention times are met (see Table G-2).
- 11.6.4 Retention times-Retention time of the internal standard. The absolute retention time of the TCB internal standard should be within the range 21.0 ± 0.5 minutes. Relative retention times of the n-alkanes: The retention times of the n-alkanes relative to the TCB internal standard shall be similar to those given in Table G-2.

12.0 Calculations

The concentration of oil in NAFs drilling fluids is computed relative to peak areas between C8 and C13 (using the Total Area Integration method) or peak areas from extracted ion profiles (using the Extracted Ion Profile Method). In either case, there is a measurable amount of peak area, even in clean drilling fluid samples, due to spurious peaks and electrometer “noise” that contributes to the total signal measured using either of the quantitation methods. In this procedure, a correction for this signal is applied, using the blank or clean sample correction technique described in American Society for Testing Materials (ASTM) Method D-3328-90, Comparison of Waterborne Oil by Gas Chromatography. In this method, the “oil equivalents” measured in a blank sample by total area gas chromatography are subtracted from that determined for a field sample to arrive at the most accurate measure of oil residue in the authentic sample.

12.1 Total Area Integration Method

- 12.1.1 Using C8 to C13 TIC area, the TCB area in the clean NAF sample and the TIC linear regression curve, compute the oil equivalent concentration of the C8 to C13 retention time range in the clean NAF. Note: The actual TIC area of the C8 to C13 is equal to the C8 to C13 area minus the area of the TCB.
- 12.1.2 Using the corresponding information for the authentic sample, compute the oil equivalent concentration of the C8 to C13 retention time range in the authentic sample.
- 12.1.3 Calculate the concentration (% oil) of oil in the sample by subtracting the oil equivalent concentration (% oil) found in the clean NAF from the oil equivalent concentration (% oil) found in the authentic sample. The C8 to C13 TIC area will not work well for clean NAF samples that contain measurable amounts of paraffins in the C8 to C13 range.

12.2 EIP Area Integration Method

- 12.2.1 Using the ratio of the 105 EIP area to the TCB m/z 91 EIP area in the clean NAF sample, and the appropriate EIP linear regression curve, compute the oil equivalent concentration of the in the clean NAF.
- 12.2.2 Using the corresponding information for the authentic sample, compute its oil equivalent concentration.
- 12.2.3 If the ratio of the of the 105 EIP area to the TCB m/z 91 EIP area for the authentic sample is greater than that for the 1% formation oil equivalent calibration standard, the sample is considered contaminated with formation oil.

13.0 Method Performance

13.1 Specification in this method are adopted from EPA Method 1663, Differentiation of Diesel and Crude Oil by GC/FID (Reference 16.4).

13.2 Single laboratory method performance using an Internal Olefin (IO) drilling fluid fortified at 0.5% oil using a 35 API gravity oil was:

Precision and accuracy $94 \pm 4\%$

Accuracy interval-86.3% to 102%

Relative percent difference in duplicate analysis-6.2%

14.0 Pollution Prevention

14.1 The solvent used in this method poses little threat to the environment when recycled and managed properly.

15.0 Waste Management

- 15.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restriction, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.
- 15.2 All authentic samples (drilling fluids) failing the RPE (fluorescence) test (indicated by the presence of fluorescence) shall be retained and classified as contaminated samples. Treatment and ultimate fate of these samples is not outlined in this SOP.
- 15.3 For further information on waste management, consult "The Waste Management Manual for Laboratory Personnel", and "Less is Better: Laboratory Chemical Management for Waste Reduction", both available from the American Chemical Society's Department of Government Relations and Science Policy, 1155 16th Street NW, Washington, D.C. 20036.

16.0 References

- 16.1 Carcinogens-"Working With Carcinogens." Department of Health, Education, and Welfare, Public Health Service, Centers for Disease Control [available through National Technical Information Systems, 5285 Port Royal Road, Springfield, VA 22161, document no. PB-277256]: August 1977.
- 16.2 "OSHA Safety and Health Standards, General Industry [29 CFR 1910], Revised." Occupational Safety and Health Administration, OSHA 2206. Washington, DC: January 1976.
- 16.3 "Handbook of Analytical Quality Control in Water and Wastewater Laboratories." USEPA, EMSSL-CI, EPA-600/4-79-019. Cincinnati, OH: March 1979.
- 16.4 "Method 1663, Differentiation of Diesel and Crude Oil by GC/FID, Methods for the Determination of Diesel, Mineral, and Crude Oils in Offshore Oil and Gas Industry Discharges, EPA 821-R-92-008, Office of Water Engineering and Analysis Division, Washington, DC: December 1992.

ATTACHMENT 1 NOTICE OF INTENT INFORMATION

Attachment 1

Notice of Intent



FILE NUMBER _____ (for ADEC use)

NOTICE OF INTENT (NOI) / APPLICATION TO DISCHARGE UNDER:

APDES General Permit No. AKG315100

For Oil and Gas Exploration Facilities in State Waters in Cook Inlet

Please submit this NOI to:

ALASKA DEPARTMENT OF ENVIRONMENTAL CONSERVATION

Wastewater Discharge Authorization Program

555 Cordova Street

Anchorage, Alaska 99501

Submittal of this document constitutes notice that the party identified in Section 3 intends to be covered by the APDES General Permit No. AKG315100 authorizing discharges into waters of the United States resulting from oil and gas exploration and obligates the applicant to comply with the terms and conditions of the permit. Please provide all information below per each site proposed for exploration activities. Attach supplemental information sheets as appropriate.

SECTION 1 – PERMIT INFORMATION

Previous Permit or Authorization No. (if applicable):

Please describe the coverage requested.

- ☐ New Use: A wastewater discharge that has not been authorized under a previous permit, including new facilities.
- ☐ New Use: A wastewater discharge that was previously authorized under an Individual Permit or a different General Permit.
- ☐ Reissuance: A wastewater discharge that was previously authorized under the 2007 Permit.

SECTION 2 – FACILITY INFORMATION

Facility Name:

Phone:

Street/Location:

Fax:

City (nearest city if not in a city):

State: Alaska

Zip:

Email Address:

February 25, 2013

| | | |
|--|---------------|------------------|
| SECTION 2 – FACILITY INFORMATION (Continued) | | |
| Population Served by this Facility: | | |
| Daily discharge Flow Rate: (PERMITTED) | | |
| Average: | Maximum: | Design Capacity: |
| SECTION 3 – RESPONSIBLE PARTY INFORMATION (Owner/Operator or Person responsible for overall management of the project and discharge) | | |
| First Name: | Last Name: | Phone: |
| Title: | | |
| Mailing Address: | | Fax: |
| City: | State: | Zip: |
| E-mail Address: | | |
| SECTION 4 –ON-SITE CONTACT/OPERATOR INFORMATION [] Check if same as Responsible Party | | |
| First Name: | Last Name: | Phone: |
| Title: | | |
| Mailing Address: | | Fax: |
| City: | State: Alaska | Zip: |
| E-mail Address: | | |
| SECTION 5 – BILLING INFORMATION | | |
| First Name: | Last Name: | Phone: |
| Title: | | |
| Mailing Address: | | Fax: |
| City: | State: Alaska | Zip: |
| E-mail Address: | | |

SECTION 6 – RECEIVING WATER INFORMATION

Name of Receiving Waterbody or Area:

Exploration Facilities are required to designate the sites where they will be operating. Authorizations will be issued per site location. Provide detailed information and vicinity maps on proposed discharge locations.

Do you wish to request to operate in a particular geographical area? ☐ Yes ☐ No

If you answered yes to the above question, please provide a map and description of the area of coverage and the latitude and longitude of the initial location of the facility.

End of outfall location:

Facility Latitude / Longitude in **either**
decimal degrees **or** in *degrees: minutes: seconds*:

Approximate First

Location:

Beginning Date

of Operation:

Expected Duration

of Operation:

Seasonal Discharger: ☐ Yes ☐ No If you answered yes, please provide the requested months of the proposed discharge

Latitude: Longitude: Determined by: ☐ PERMITS ☐ Map ☐ Internet

SECTION 7 - REQUEST FOR MIXING ZONE AND EFFLUENT MODIFICATION FROM DEC

Do you wish to request a mixing zone from DEC? ☐ Yes ☐ No

If you answered “No” to the above question or have questions concerning mixing zones, please contact the domestic wastewater permitter at the DEC office closest to your facility.

Anchorage area 907-269-6285; Fairbanks area 907-451-2183; Juneau area 907-465-5180

THE FOLLOWING INFORMATION MUST BE PROVIDED IF REQUESTING A MIXING ZONE AND YOU ANSWERED “YES” IN SECTION 7. The burden of proof for justifying a mixing zone through demonstrating compliance with the requirements of 18 AAC 70.240 – 18 AAC 70.270 rests with the applicant. Data from late winter/early spring and late summer/early fall is preferable.

Distance from discharge

to shoreline:

Number of ports

and spacing:

Depth of discharge (“zero” for surface discharges:

Diameter of

port or ports:

Length of diffuser:

Uses of Receiving Water at Distance from Diffuser or End of Pipe

| USE | DISTANCE | UNITS |
|-----|----------|-------|
|-----|----------|-------|

| | | |
|---------------------------|--|--|
| Supply for drinking water | | |
|---------------------------|--|--|

SECTION 7 - REQUEST FOR MIXING ZONE AND EFFLUENT MODIFICATION FROM DEC (Continued)

| USE | DISTANCE | UNITS |
|--|----------|-------|
| Supply for agriculture including irrigation & stock water | | |
| Supply for aquaculture | | |
| Supply for industrial use | | |
| Contact recreation | | |
| Secondary recreation | | |
| Fish spawning | | |
| Harvesting and consumption of raw fish of other aquatic life | | |

SECTION 8 – ADDITIONAL INFORMATION TO INCLUDE

SITE MAP: Submit a site map showing the exact location (latitude and longitude) of all facilities associated with the project. Include a topographic map or aerial photograph showing the general location of the facility, the expected flow direction of the discharge, and discharge area.

FOR NEW OR REVISED OPERATIONS: Provide a brief description of the treatment process(es) provided by the facility including the level of treatment and type of disinfection (if any). Include schematic flow diagram of the wastewater treatment process. If available, please provide the past year's worth of monitoring data.

ENGINEERED PLAN APPROVAL: Provide either proof of approval by DEC or the submission of plans to DEC for the system and all associated facilities, as required by 18 AAC 72.205, 72.200, and 72. 600.

SECTION 9 - REQUEST FOR A WAIVER FROM MINIMUM TREATMENT REQUIREMENTS:

In accordance with 18 AAC 72.050(d)(1) – (5) and 18 AAC 72.060(b), an applicant seeking a waiver from the minimum treatment requirements of 18 AAC 72.050(a)(1) or (a)(4) shall submit a report prepared by a registered engineer, for approval by DEC. The report shall:

- (1) Explain how public health, public and private water systems, and the environment will be adequately protected with the reduced level of treatment proposed;
- (2) Describe the volume, characteristics, frequency, and duration of the discharge;
- (3) Include the plans required by 18 AAC 72.200 and/or 18 AAC 72.600;
- (4) Identify water and existing or potential drinking water sources within 200 feet of the discharge area; and
- (5) Describe any other environmental factor that is important in approving the lesser treatment level, including:
 - (a) The hydrological characteristics of the receiving water, including flushing ability, tide, and current;
 - (b) The local topographic, geologic, and soil characteristics; and
 - (c) Existing and potential uses of the water, including drinking, aquaculture, food processing, food gathering, fishing, boating, swimming, and recreation.

SECTION 10 – CERTIFICATION

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

Signature

Title

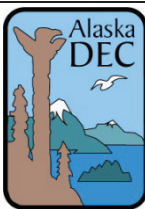
Printed Name

Date

ATTACHMENT 2 NONCOMPLIANCE NOTIFICATION

Attachment 2

Noncompliance Notification



Alaska Department of Environmental Conservation

Division of Water, Compliance and Enforcement Program

555 Cordova Street

Anchorage, Alaska 99501

Nationwide Toll Free: 1(877) 569-4114 Anchorage/International: (907) 269-4114

Fax: (907) 269-4604 E-mail address: dec-wqreporting@alaska.gov.

NONCOMPLIANCE NOTIFICATION

| | | | |
|---|---|--|--|
| GENERAL INFORMATION | | PERMIT# (if any): | |
| Owner or Operator: | Facility Name: | Facility Location: | |
| Person Reporting: | Phone Numbers of Person Reporting: | Reported How? (e.g. by phone): | |
| Date/Time Event was Noticed: | Date/Time Reported: | Name of DEC Staff Contacted: | |
| VERBAL NOTIFICATION MUST BE MADE TO ADEC WITHIN 24 HOURS OF DISCOVERY OF NONCOMPLIANCE | | | |
| INCIDENT DETAILS (attach additional sheets, lab reports, and photos as necessary) | | | |
| Period of Noncompliance | Start Date/Time (exact): | End Date/Time (exact): | |
| If noncompliance has not been corrected, provide a statement regarding the anticipated time the noncompliance is expected to continue: | | | |
| Estimated Quantity involved (volume or weight): | | | |
| Description of the noncompliance and its cause (be specific): | | | |
| Actions taken to reduce, eliminate, and prevent reoccurrence of noncompliance and Actual/Potential Impact on Environmental Health (describe in detail) (e.g. Supplied drinking water to nearby well owners and informed well owners not to drink from wells until further notice) | | | |
| Permit Condition Deviation (Identify each permit condition exceeded during the event.) | | | |
| <u>Parameter (e.g. BOD pH)</u> | <u>Permit Limit</u> | <u>Exceedance (sample result)</u> | <u>Sample Date</u> |
| Corrective Actions (Attach a description of corrective actions taken to restore the system to normal operation and to minimize or eliminate chances of recurrence.) | | | |
| Environmental Damage: (if yes, provide details below) | | <input type="checkbox"/> Yes | <input type="checkbox"/> No <input type="checkbox"/> Unknown |
| Actual /Potential Impact on Environment/Public Health (describe in detail) | | | |
| I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations. | | | |
| Name: | Title: | Signature: | Date: |
| FORMS MUST BE SENT TO ADEC WITHIN FIVE DAYS OF BECOMING AWARE OF THE EVENT. | | | |